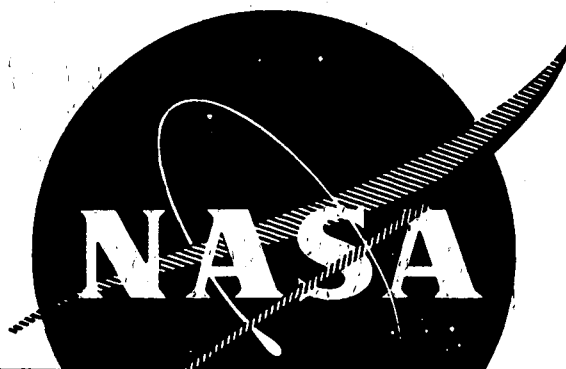


g-p
mix

NAS CR 121099

TRW ER-7624



(NASA-CR-121099) DEVELOPMENT OF A POWER
METALLURGY SUPERALLOY FOR USE AT
1800-2000 F (980-1090 C) Final Report,
11 May 1970 - 20 Sep. 1973 (TRW
Equipment Labs.) 98 p HC \$7.00 CSCL 11F

N73-18537

Unclas
63443

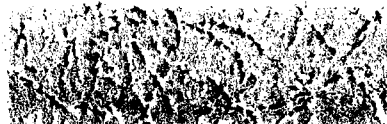
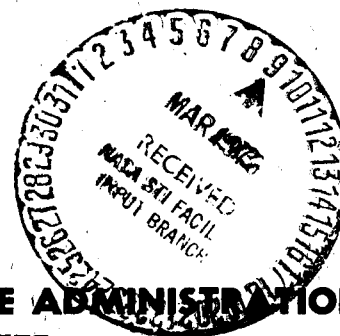
G3/17

DEVELOPMENT OF A POWDER METALLURGY SUPERALLOY FOR USE AT 1800-2000° F (980-1090° C)

FINAL REPORT

Prepared for

**NATIONAL AERONAUTICS AND SPACE ADMINISTRATION
LEWIS RESEARCH CENTER
UNDER CONTRACT NAS 3-13488**



TRW MATERIALS TECHNOLOGY LABORATORIES

CLEVELAND, OHIO

99

NOTICE

This report was prepared as an account of Government sponsored work. Neither the United States, nor the National Aeronautics and Space Administration (NASA), nor any person acting on behalf of NASA:

- A.) Makes any warranty or representation, expressed or implied, with respect to the accuracy, completeness, or usefulness of the information contained in this report, or that the use of any information, apparatus, method, or process disclosed in this report may not infringe privately owned rights; or**
- B.) Assumes any liabilities with respect to the use of, or for damages resulting from the use of any information, apparatus, method or process disclosed in this report.**

As used above, "person acting on behalf of NASA" includes any employee or contractor of NASA, or employee of such contractor, to the extent that such employee or contractor of NASA, or employee or such contractor prepares, disseminates, or provides access to, any information pursuant to his employment or contract with NASA, or his employment with such contractor.

- a -

1. Report No. 121899 NASA-CR-1210 99 99		2. Government Accession No.		3. Recipient's Catalog No.	
4. Title and Subtitle DEVELOPMENT OF A POWDER METALLURGY SUPERALLOY FOR USE AT 1800-2000°F (980-1090°C)				5. Report Date February 15, 1973	
				6. Performing Organization Code	
7. Author(s) Charles S. Kortovich				8. Performing Organization Report No. TRW ER-7624	
				10. Work Unit No.	
9. Performing Organization Name and Address TRW Inc. TRW Equipment 23555 Euclid Avenue Cleveland, Ohio 44117				11. Contract or Grant No. NAS-3-13488	
				13. Type of Report and Period Covered Contractor Report May 11, 1970 - Sept. 20, 1973	
12. Sponsoring Agency Name and Address National Aeronautics and Space Administration Washington, D.C. 20546				14. Sponsoring Agency Code	
15. Supplementary Notes Project Manager, Fredric H. Harf, Materials Structures Division, NASA Lewis Research Center, Cleveland, Ohio 44135					
16. Abstract A program was conducted to develop a powder metallurgy nickel-base superalloy for 1800-2000°F (980-1090°C) temperature applications. The feasibility of a unique concept for alloying carbon into a superalloy powder matrix and achieving both grain growth and a discrete particle grain boundary carbide precipitation was demonstrated. The process consisted of blending metastable carbides with a carbon free base alloy and consolidating this blend by hot extrusion. This was followed by heat treatment to grow a desired ASTM No. 2-3 grain size and to solution the metastable carbides to allow precipitation of discrete particle grain boundary carbides during subsequent aging heat treatments. The best alloy developed during this program was hydrogen-atomized, thermal-mechanically processed, modified MAR-M246 base alloy plus VC (0.28 w/o C). Although below those for cast MAR-M246, the mechanical properties exhibited by this alloy represent the best combination offered by conventional powder metallurgy processing to date. The elimination of three microstructural defects should provide a considerable increase in the property levels obtained in this program. These include incomplete compaction, inconsistent grain growth response, and a large embrittling angular carbide resulting from the decomposition of the original blended metastable carbide.					
17. Key Words (Suggested by Author(s)) Nickel Alloys Powder Metallurgy Turbine Blade Applications Thermomechanical Processing MAR-M246 Metastable Carbides				18. Distribution Statement Unclassified - Unlimited	
19. Security Classif. (of this report) Unclassified		20. Security Classif. (of this page) Unclassified		21. No. of Pages 93	
				22. Price* \$37.00	

FOREWORD

The work described in this report was performed in the Materials Technology Laboratory of TRW Inc. under the sponsorship of the National Aeronautics and Space Administration, Contract NAS-3-13488. The purpose of the study was the development of powder metallurgy superalloy for use at 1800-2000°F (980-1090°C). The program was administered for TRW Inc. by Dr. H. E. Collins, Program Manager. The principal investigator was C. S. Kortovich, with technical assistance provided by W. J. Yankauskas, W. J. Lee, and J. Laughlin. The NASA Program Manager was F. H. Harf, the Research Advisor R. V. Miner, both of the Materials and Structures Division.

Prepared by: *C. S. Kortovich*
C. S. Kortovich

Reviewed by: *H. E. Collins*
H. E. Collins
Principal Engineer

Approved by: *J. A. Alexander*
J. A. Alexander
Manager
Materials Research Department

AL

TABLE OF CONTENTS

	<u>Page</u>
I. SUMMARY	1
II. INTRODUCTION	3
III. PROGRAM OUTLINE FOR THE DEVELOPMENT OF A HIGH TEMPERATURE POWDER METALLURGY NICKEL-BASE SUPERALLOY	6
A. Task I - Powder Production and Consolidation	6
B. Task II - Property Optimization	7
IV. TASK I - POWDER PRODUCTION AND CONSOLIDATION	8
A. Materials and Procedures	8
1. Superalloy Powder Production	8
2. Powder Consolidation	8
3. Extrusion Bar Inspection	10
4. Mechanical Property Evaluations	12
B. Results and Discussion	12
1. Radiographic and Chemical Analysis	12
2. Metallographic Analysis	12
3. As-Extruded Mechanical Properties	16
V. TASK II - PROPERTY OPTIMIZATION	21
A. Series I - Selection of Most Desirable Metastable Carbide	21
1. Materials and Procedures	21
a. Heat Treatment Studies	21
b. Mechanical Property Evaluations	21
c. Vacuum Wash Procedure Change	21
2. Results and Discussion	23
a. Argon Atmosphere Solution Heat Treatments	23
i. Single Exposure Solution Heat Treatments	23
ii. Multiple Exposure Solution Heat Treatments	31
b. Solution Heat Treatments in Vacuum	39
c. Vacuum Washed Base Alloy	39
d. Mechanical Property Results	44
i. High Temperature Property Results	44
ii. Intermediate Temperature Properties	51
3. Summary	51

llh

TABLE OF CONTENTS (continued)

	<u>Page</u>
B. Series 2 - Optimization of Carbon Content	54
1. Materials and Procedures	54
a. Powder Production	54
b. Powder Consolidation	54
c. Property Optimization Procedures	57
2. Results and Discussion	58
a. Extrusion Bar Inspection	58
i. Radiographic and Chemical Analysis	58
ii. Metallographic Analysis	60
b. Solution and Grain Growth Heat Treatment Study.	60
c. Thermal Mechanical Process Study	64
d. Mechanical Property Results	65
e. Metallographic Analysis of Failed Test Bars	69
3. Summary	77
C. Series 3 - Elimination of Microstructural Defects	82
1. Materials and Procedures	82
2. Results and Discussion	83
a. Radiographic and Chemical Analysis	83
b. Solution and Grain Growth Heat Treatment Study.	83
c. Mechanical Property Results	83
d. Metallographic Analysis of Failed Test Bars	86
VI. CONCLUSIONS	88
VII. RECOMMENDATIONS FOR FUTURE WORK	90
VII. REFERENCES	92

NV

I SUMMARY

The objective of this program was to develop a superalloy from powder for use as an airfoil material at temperatures from 1800°F (980°C) to 2000°F (1090°C) in high performance gas turbines. The goals of this program included the development of a nickel-base alloy with a density of less than 0.310 lbs/in³ (8.5 g/cm³) which has a tensile and stress rupture life exceeding current wrought and cast alloys. In addition, the tensile reduction of area from 1300°F (705°C) to 2000°F (1090°C) should exceed 15 percent and at room temperature should exceed 5 percent after prolonged elevated temperature exposure. The program was performed in two tasks, Task I consisting of powder production and consolidation, Task II consisting of property optimization.

The base alloy composition selected for study was a modified MAR-M246 containing 0.6 percent hafnium, 0.5 percent additional tantalum and no carbon. To obtain the beneficial effects of MC carbides of hafnium and tantalum, carbon was added to the base composition in the form of metastable carbides. In the first series of alloys studied in the program, argon atomized powder was used as the base alloy. Three carbides with a range of thermodynamic stabilities were evaluated to determine which has sufficient stability to survive the hot extrusion consolidation process intact, go into solution during grain growth heat treatments and precipitate hafnium and tantalum rich carbides during final aging heat treatments. The added carbides were Cr₃C₂, VC, and TiC. The VC and TiC were selected as the best for grain growth, carbide precipitation, stress rupture, and tensile properties. Argon-atomized powder was not utilized for the final two series of alloys for the program because of the amount of gas porosity produced in the microstructure as a result of the high temperature heat treatments required for grain growth. Hydrogen-atomized powder was utilized for the remainder of the program.

A second series of alloys containing VC was evaluated to optimize the carbon content. Also included in this series was an alloy containing TiC at the same carbon level as used in the first alloy series, to compare the resultant mechanical properties of hydrogen versus argon atomized powder. It was established that no gas porosity existed in heat treated bar stock made from hydrogen-atomized powder and that VC additions at the 0.28 w/o carbon level provided the best combination of high temperature properties. Thermal-mechanical processing which aided in providing a more uniformly coarsened grain size was utilized to obtain 1900°F (1038°C)/15 ksi (104 MN/m²) stress rupture properties approximately 52 percent of the cast value and tensile properties ranging from 75-90 percent of the cast values.

Metallographic analysis of the failed test specimens isolated three problem areas which still must be overcome to improve property levels. First, the blending operation used to mix the metastable carbides with the base powder did not produce a homogeneous distribution throughout the microstructure. The inhomogeneities resulted in variations in heat treatment response from specimen to specimen which caused premature rupture failures because of the large number of small grains located throughout the microstructure. Second,

incomplete compaction during the consolidation of relatively coarse (-60 +325 mesh size) hydrogen-atomized powder left areas of porosity throughout the specimens. This porosity had an irregular, elongated shape as opposed to the spherical gas porosity observed with argon-atomized powders. Third, an angular carbide resulting from the decomposition of the original blended carbides was observed to contribute to premature failure by initiating cracks. It was possible, however, to mitigate the crack initiating tendency of this microconstituent by low temperature aging heat treatments.

II INTRODUCTION

The desire for faster and larger aircraft has given impetus to the improvement of existing materials as well as to the development of new materials capable of maintaining their strength and their creep, oxidation, and thermal fatigue resistance at high temperatures. In response to these needs considerable effort has been expended on the nickel-base superalloy system. As temperature levels were increased and the workability of wrought alloys became severely limited, investment casting processes have emerged for the production of complex turbine blade configurations. Alloy development programs utilizing investment casting techniques have resulted in complex alloys represented by MAR-M246 and TRW-NASA VIA. In these complex alloys with larger amounts of more alloying additions castability has become a serious problem and the degree of segregation has tended to increase. Alloy segregation has led to undesirable scatter in the mechanical properties.

The utilization of ultrapure fine prealloyed superalloy powders offers the potential to limit this segregation to a microscopic scale and offers new alloying innovations by permitting the formulation of compositions which can not be achieved by conventional techniques. Through powder metallurgy techniques the microstructure can also be controlled to a greater degree than is possible in cast materials, i.e., the matrix of the alloy can be strengthened independently of the strengthening being accomplished in the grain boundary regions. The use of powders for high temperature superalloys is not without problems. In order to develop high temperature strength, sophisticated process control is required to minimize interstitial impurities. In addition, void elimination, a discrete particle grain boundary carbide morphology, and an optimum grain size are also required to develop the proper combination of high temperature strength and ductility.

To date, conventional processing of prealloyed powders whose compositions are similar to those of cast alloys has not achieved comparable 1800°F (980°C) - 2000°F (1090°C) tensile and stress rupture properties. The primary problem has been the inability to achieve grain sizes (ASTM 2-3) comparable to those of the cast alloys. Grain growth has been achieved in modified alloys with reduced carbon content, but this at a sacrifice in the grain boundary strengthening offered by carbon. The unusual situation exists wherein the important carbide strengthening particles are themselves responsible for pinning grain boundaries and retarding the desired grain growth so critical to high temperature properties.

Mechanical alloying, a recent unconventional powder metallurgy approach, employing dispersion strengthening has achieved stress rupture properties similar to those of today's casting alloys although with lower tensile strengths. Developed in low alloy content superalloy powders this technique offers the unique combination of dispersion hardening for high temperature applications and gamma-prime strengthening for intermediate temperature usage. A serious limitation to the eventual production scale-up of this process may be the fact that property levels have been achieved only through hot extrusion consolidation.

Unless a compaction technique offering shapes closer to final dimensions can be developed, parts would have to be machined from extruded bar stock. This would negate the important powder metallurgy advantage of optimum materials utilization.

This program was initiated to develop a superalloy from powder for use as an airfoil material at temperatures from 1800 to 2000°F (980 to 1090°C) in high performance gas turbines. The goals of this program included the development of a nickel-base alloy with a tensile strength and stress rupture life exceeding that of the current cast alloys and a density of less than 0.310 lbs/in³ (8.58 g/cm³). The target properties for the material are a 100 hour stress rupture life at 1900°F (1038°C) under a stress of 15,000 psi (104 MN/m²). In addition, the tensile reduction of area from 1300 to 2000°F (704 to 1090°C) should exceed 15 percent and at room temperature should exceed 5 percent after prolonged elevated temperature exposure. This alloy should also be compatible with protective coatings and should be amenable to diffusion bonding.

The approach taken in this program to achieve the target goals was considerably different from that utilized in previous powder metallurgy studies which involved the duplication of casting alloy chemistries. Utilization of the prealloyed powders technique has been unsatisfactory because of the detrimental effect of carbides upon the ability of powder metallurgy material to attain sufficiently large grain size. Instead of utilizing prealloy powders containing increased amounts of alloy strengtheners, an internal carburization approach was used in conjunction with chemical modifications of a cast nickel-base superalloy for this program. Carbon free prealloy powders of the base chemistry were blended with metastable carbides and consolidated by hot extrusion. Heat treatments were then applied to solution the metastable carbides, thereby permitting grain growth to occur and releasing carbon for subsequent precipitation as discrete particle grain boundary carbides.

The program was divided into two tasks. Task I consisted of powder production and the consolidation of the alloy compositions for the initial screening studies. Argon gas atomization was utilized to produce superalloy powder with a low interstitial content. A base alloy having the maximum amount of alloying while still meeting the program density requirements was selected for the program. Its composition was a variation of MAR-M246 without the carbon and with 0.6 percent hafnium and 0.5 percent additional tantalum. To obtain the beneficial effects of MC carbides of hafnium and tantalum, metastable carbides Cr₃C₂, VC, and TiC were blended with the carbon free master powder. After solutioning and grain growth heat treatments, the MC hafnium and tantalum rich carbides were precipitated along preferred grain boundary sites.

Task II entailed a screening study and property optimization of the consolidated powder alloys. Four alloys, including the carbon free base, were evaluated for mechanical properties including room temperature, 1400°F (760°C), and 1900°F (1038°C) tensile and 1900°F (1038°C)/15 ksi (104 MN/m²) stress rupture properties. The best carbide for use as a carbon source was selected on the

basis of mechanical properties, resultant grain growth and MC carbide precipitation. It was then evaluated at two different levels to determine the optimum carbon content in the alloy. In addition to this, Task II also included an evaluation of the hydrogen gas powder atomization process and a double consolidation process consisting of hot pressing plus hot extrusion.

This report summarizes the results obtained on NASA Contract NAS3-13488. It includes a review of the program to develop a high temperature nickel-base alloy from powder, a review of the experimental procedures, a summary of the experimental results, a discussion of these results, and recommendations for future work to improve the property levels attained during the program.

III PROGRAM OUTLINE FOR THE DEVELOPMENT OF A HIGH TEMPERATURE POWDER METALLURGY NICKEL-BASE SUPERALLOY

The development of high temperature properties in the powder product requires grain growth through the use of high temperature thermal treatments and precipitation of a discrete particle grain boundary type morphology through the use of low temperature aging treatments. This can be accomplished by adding carbon in the form of metastable carbides which decompose during grain growth heat treatments and will not impair the growth of the grain themselves. Carbon is released for subsequent precipitation as MC type hafnium and tantalum rich carbides to enhance strength and ductility. The base alloy selected for the program was a modification of MAR-M246 without carbon and with 0.6 w/o (weight percent) hafnium and an additional 0.5 w/o tantalum. In order to internally carburize this alloy, three metastable carbides were evaluated during the program, Cr_3C_2 , TiC , and VC .

Carbides with different stabilities were evaluated because the kinetics of carbide solutioning and precipitation were not quantitatively known. A range of stability was investigated so that a carbide which decomposes quickly could form hafnium and tantalum rich carbides before grain growth could occur, while a stable carbide would not decompose and produce the desired MC carbides. The free energy of formation of the pure carbides at 1880°F (1300°C) are included in the following table per mole of carbon (1):

<u>Reaction</u>	<u>Free Energy</u>
$\text{Ti} + \text{C} = \text{TiC}$	-40,490 cal/mole
$\text{V} + \text{C} = \text{VC}$	-22,150 cal/mole
$3/2\text{Cr} + \text{C} = 1/2\text{Cr}_3\text{C}_2$	-11,830 cal/mole

The program for the development of a high temperature nickel-base powder superalloy was divided into two tasks. Task I consisted of powder production and consolidation. Task II consisted of a property optimization effort in which various thermal-mechanical treatments were employed to improve the high temperature properties of the powder product.

A. Task I - Powder Production and Consolidation

Material for the first property optimization studies (alloy Series I) was argon atomized and shipped to TRW under a protective covering of argon gas. Four alloys, including the carbon free base composition, were canned under an argon atmosphere, evacuated, and single extruded at a 16:1 reduction ratio at 2175°F (1135°C). The three alloys containing the metastable carbides were at the 0.20 w/o C level. Radiographic, chemical, and metallographic analysis completed the evaluation of the first four extrusions.

B. Task II - Property Optimization

The initial objective of this portion of the program was to determine which metastable carbide would go into solution during high temperature heat treatments without inhibiting grain growth and would permit the desired hafnium and tantalum rich MC carbides to form in the grains and in the grain boundaries after subsequent aging heat treatments. On the basis of room temperature, 1400°F (760°C) and 1900°F (1038°C) tensile and 1900°F (1038°C)/15 ksi (104 MN/m²) stress rupture tests the most desirable metastable carbide was selected to optimize the carbon content for the alloy. After high temperature solution treatments in argon, including a series of exposures at consecutively increasing temperatures to homogenize the grain boundary areas and raise the incipient melting temperatures, and vacuum heat treatments to reduce porosity observed in material heat treated in argon, both TiC and VC carbide additions were selected for further evaluation on the basis of their resultant mechanical properties.

In order to eliminate the porosity observed in the initial extrusions, hydrogen atomized base powder was utilized for the remainder of the program. The second series of extrusions comprised two alloys containing VC at the 0.15 and 0.28 w/o level to optimize the carbon content and a third containing TiC at the 0.20 w/o level to evaluate the resultant mechanical properties of argon versus hydrogen atomized powder. Side press forging plus subsequent solution and aging heat treatments were utilized to improve the properties of these alloys. Subsequent mechanical property tests indicated that the base alloy plus VC at the 0.28 w/o carbon level exhibited the best combination of high temperature properties.

Further work was performed with this alloy with VC at the 0.28 w/o carbon level to eliminate areas of incomplete compaction observed in the second series of extrusions and to alter the crack initiating tendencies of an angular MC type carbide produced by the decomposition of the original blended carbides. A double compaction technique consisting of hot pressing followed by hot extrusion was used instead of single extrusion, and a 100 hour age at 1700°F (925°C) was used to round the sharp corners of the angular carbides.

IV TASK I - POWDER PRODUCTION AND CONSOLIDATION

A. Materials and Procedures

1. Superalloy Powder Production

The starting material for this program was 195 pounds (89 kg) of vacuum melted master metal of the modified MAR-M246 composition purchased from the Cannon-Muskegon Corporation, Michigan, and shipped to the Federal-Mogul Corporation, Ann Arbor, Michigan, where it was argon gas-atomized. The yield from this master metal was 136 pounds (62 kg) -100 mesh powder, 49 pounds (22 kg) of +100 mesh powder and 11 pounds (6 kg) of unused ingot (Cannon-Muskegon heat number VE-103). The powder was shipped to TRW under a protective argon atmosphere. The particle shape of the argon atomized powders was nearly perfectly spherical, Figure 1, and the microstructure was similar to that of a normally cast superalloy except that the dendrite size and, hence, interdendritic spacing was much finer in the rapidly quenched powder than in a conventional casting. Many fine particles were observed attached to the primary powder particles and a number of spherical voids were seen within the powders themselves.

2. Powder Consolidation

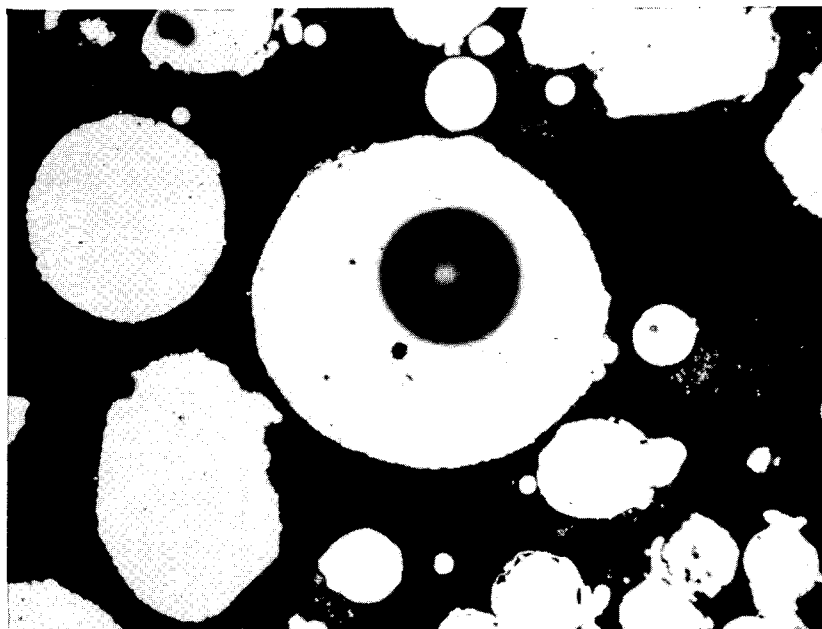
For the first series of alloys the metastable carbides were blended with the master powder in an inert argon atmosphere. Approximately 8 pounds (3.6 kg) of powder were prepared for each of the 4 extrusions by adding the appropriate amount of metastable carbide to the -100 mesh base powder as listed below in Table 1 to reach a nominal carbon content of 0.20 w/o.

Table 1

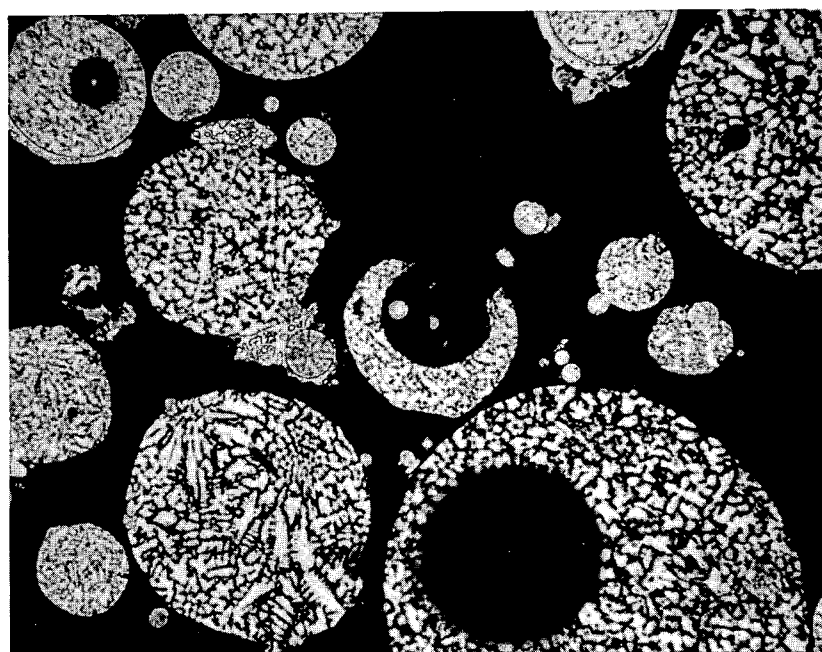
Constituents of First Four Extrusions Made in First Alloy Series

<u>Extrusion No.</u>	<u>Amount -100 Mesh Base Alloy (g)</u>	<u>Amount Carbide (g)</u>	<u>Percentage Carbon in Carbide (w/o)</u>	<u>Carbon Content of Alloy (w/o)</u>
1	3630	-	-	0
2	3575.7	54.3Cr ₃ C ₂	13.2	.20
3	3592.8	36.0Ti	19.5	.20
4	3592	38VC	18.3	.20

The alloy constituents were placed in a TIG welding chamber along with a double conical blender. The TIG chamber was back filled with argon and the proper amount of 6-8 micron Cr₃C₂, 3-6 micron TiC, or 3-4 micron VC was sealed in a Simmons-Kelly conical blender along with the base powder. The blender was



(a) Unetched



(b) Etched

Figure 1. Light photomicrographs of as-received argon atomized superalloy powder. 250X

This page is reproduced at the back of the report by a different reproduction method to provide better detail.

then removed from the TIG chamber, mounted on a lathe and rotated for one hour at 50 rpm before transfer into the TIG chamber along with the 304 stainless steel extrusion can, Figure 2.

After the TIG chamber was again back filled with argon, the 8 pounds (3.6 kg) of powder were removed from the blender and placed into the extrusion can (approximately 75 percent dense) and covered with a 16 gage (1.5 mm) 304 stainless baffle. After pumping down overnight to a vacuum of approximately 0.2 microns (2.66×10^{-2} N/m²), the chamber was back filled with argon and the nose of the extrusion can welded into place. The extrusion can was then transferred to a Sciaky Electron Beam welder where a hole was made in the nose and the chamber pumped down overnight to a vacuum of 0.1 micron or less (1.33×10^{-2} N/m²). As the gas escaped during pump-down the baffle prevented powder from being pulled out through the hole. After evacuation the electron beam was defocused and the hole resealed.

The extrusion cans were then preheated to 1500°F (815°C) for one hour in a Pereny resistance element heated box furnace and transferred to a barium chloride salt bath at 2175°F (1190°C) and soaked for one hour prior to extrusion. Single extrusion was conducted in a 700 ton Loewy Hydraulic extrusion press using zirconia coated CH-2 tool steel dies at a 16:1 reduction ratio. Ram speed was approximately 2 inches (5.1 cm)/second with a breakthrough pressure of approximately 100,000 psi (694 MN/m²). Approximately 60 inches (1.5 m) of sound bar stock were obtained from each 8 pound (3.6 kg) extrusion.

3. Extrusion Bar Inspection

Following extrusion all bar stock was radiographically inspected. Each specimen was radiographed in one position, rotated 90°, and radiographed again to be sure that no defect would go undetected because of orientation. The bar stock was then cut into 3 inch (7.6 cm) sections, decanned, and submitted for a complete chemical analysis for all alloying elements and important impurities, such as Fe, Mn, Si, and S. The analyses were performed using a combination of X-ray spectrographic, spectroscopic, and wet techniques depending on the element analyzed.

Metallographic specimens were sectioned in both the longitudinal and transverse orientation to completely characterize the as-extruded microstructure. The polishing procedure included polishing on 180 to 600 grit silicon carbide papers with a water lubricant followed by final polishing consisting of hand polishing on billiard cloth with Linde A (Al₂O₃ - 3 micron size) abrasive followed by 45 minutes on the automatic Syntron polisher using gamal cloth and Linde B (Al₂O₃ - .06 micron size) abrasive. An etch consisting of 150 ml H₂O, 50 ml HCl, 25 ml HNO₃, and 1 g CuCl₂ was used to delineate the microstructural features. Light photomicrographs at 250X, 500X and 1000X magnification were taken of characteristic features of the microstructure.

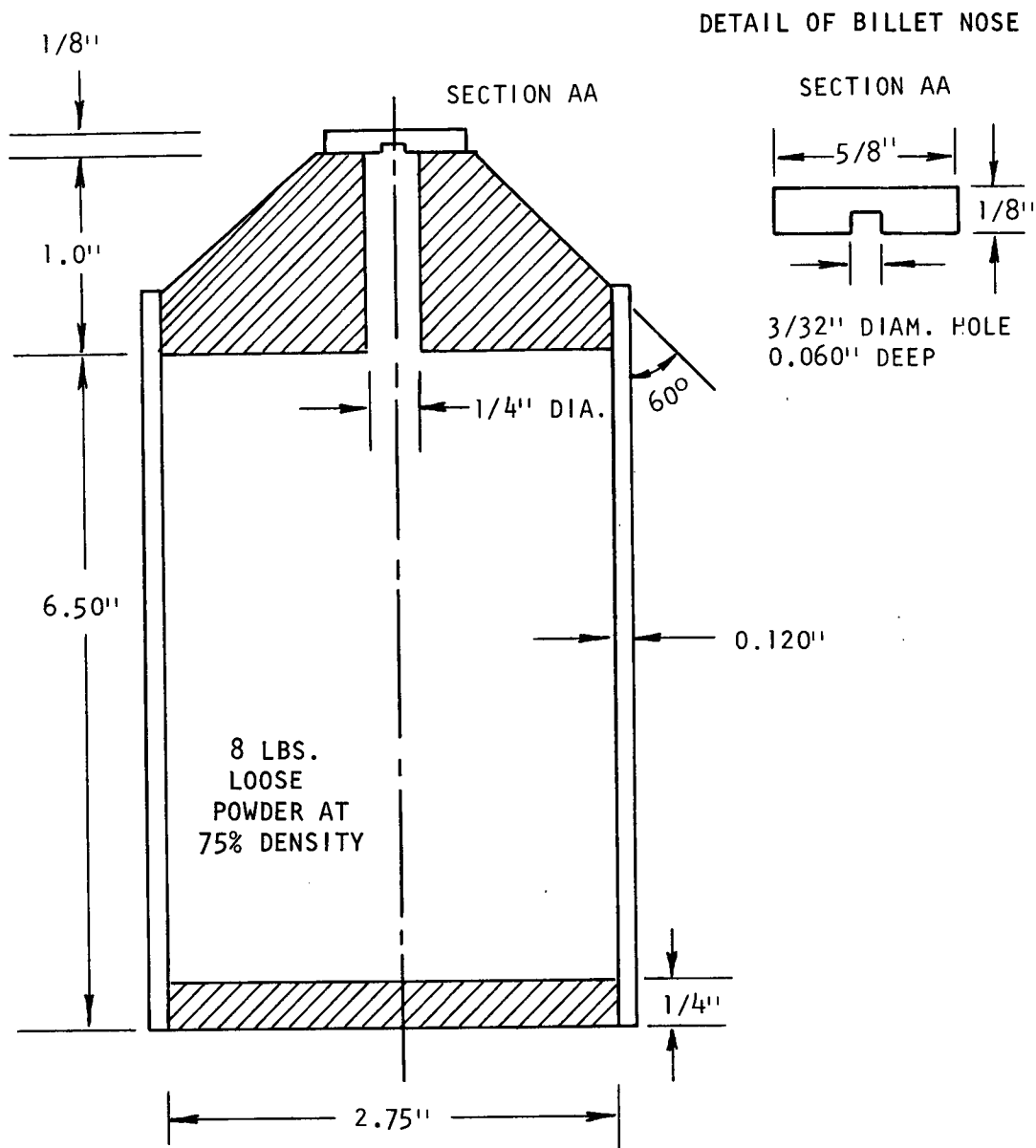


Figure 2. Schematic illustration of billet geometry for extrusion can utilized in program.

4. Mechanical Property Evaluations

Mechanical property tests were performed on material in the as-extruded condition. All testing was performed in air and included duplicate room temperature, 1400°F (760°C), and 1900°F (1038°C) tensile tests and 1900°F (1038°C)/15 ksi (104 MN/m²) stress rupture tests. The tensile tests were performed in a Model TT6 Instron Universal tensile testing machine at a cross-head speed of 0.020 in/min (0.5 mm/min.). The stress rupture tests were performed on a Model J Satec constant load creep testing machine. Light metallographic examination was conducted on all failed tensile and stress rupture bars to determine possible fracture initiation sites.

B. Results and Discussion

1. Radiographic and Chemical Analysis

All four alloys were successfully extruded and radiographic inspection indicated no visible defects. There was sufficient sound stock, approximately 60 inches (1.5 m) to conduct the required Task II optimization studies. The chemical analyses for the four extrusions are presented in Table 2 along with the original vacuum-melted billet chemistry (Cannon-Muskegon Heat No. VE-103). The attempt to incorporate approximately 0.20 w/o carbon into the alloys was successful, and the carbon levels for the various alloys were consistent. In addition to the consistency of the carbon levels, the oxygen contents of all the extrusions indicated that the program requirement of less than .01 percent (100 ppm) was also attained. For the most part, compositions were well within the program range for the aim indicated. Several exceptions were observed, however, the most noticeable being the titanium levels, which were lower than anticipated. Since TiC was added to extrusion #3 it is reasonable to expect that the titanium content would be higher than in the rest of the extrusions. These values were rechecked and found to be consistent. In addition to the low titanium values, tungsten contents were on the high side of the program range as was the molybdenum content for extrusion #3.

2. Metallographic Analysis

Metallographic analysis of the as-extruded bars revealed considerable differences among the four extrusions. Longitudinal sections of the four alloys are shown in Figure 3 at a magnification of 250X and in Figure 4 at a magnification of 1000X. The as-extruded base material evidenced recrystallization only in central portions of the bar stock and had only a limited amount of banding, primarily at the outer portions. The central portion of the bar had a grain size of ASTM No. 8 (25 microns), and while limited amounts of a gamma-prime type precipitation was observed throughout the matrix, a considerable amount was seen in the grain boundary areas.

Table 2

Composition of Billet and First Four Extrusions of First Alloy Series (w/o)

Element	Program Aim	Program Range	MAR-M246 (1)	Billet		Base + Cr ₃ C ₂ (#2)	Base + TiC (#3)	Base + VC (#4)	Method of Analysis (3)
				VE-103 (2)	Base (#1)				
C	0.20	0.18-0.22	.15	0.004	0.011	0.21	0.21	0.19	b
Cr	9.0	8.5-9.5	9.0	8.52	8.74	9.29	8.72	9.02	b
Co	10.0	9.0-11.0	10.0	9.9	10.4	10.10	10.20	10.20	a
Mo	2.5	2.0-3.0	2.5	2.56	2.86	2.86	3.15	2.98	c
W	10.0	9.4-10.6	10.0	9.72	10.7	10.7	10.6	10.7	a
Ti	1.5	1.25-1.75	1.5	1.49	1.17	1.22	1.89	1.16	a
Al	5.5	5.0-6.0	5.5	5.52	5.29	5.32	5.45	5.22	c
B	0.015	.010-.020	0.015	0.019	0.016	0.014	0.013	0.017	c
Zr	0.05	0.03-0.08	.05	0.05	0.075	0.077	0.079	0.078	c
Ta	2.0	1.7-2.3	1.5	1.91	1.88	2.13	2.22	2.08	c
Hf	0.6	0.45-0.75		0.56	0.67	0.67	0.59	0.57	b
S	<0.015	<0.015		0.007	0.003	0.001	0.001	0.001	b
O	<.01	<.01		0.0038	0.008	0.009	0.006	0.008	b
V	-	-			<.01	<.01	0.02	0.85	a
Cu	<.1	<.1			0.12	0.11	0.12	0.06	c
P	<.01	<.01			.005	.005	.005	.005	c
Fe	<.20	<.20			.05	.05	.05	.05	c
Si	<.25	<.25			0.10	0.10	0.10	0.10	c
N	<.01	<.01			0.015	0.014	0.012	0.014	b
Mn	<.1	<.1			<.05	<.05	<.05	<.05	c

(1) International Nickel Data Brochure, 1964 Edition.

(2) Cannon-Muskegon Billet

(3) Method of Analysis:

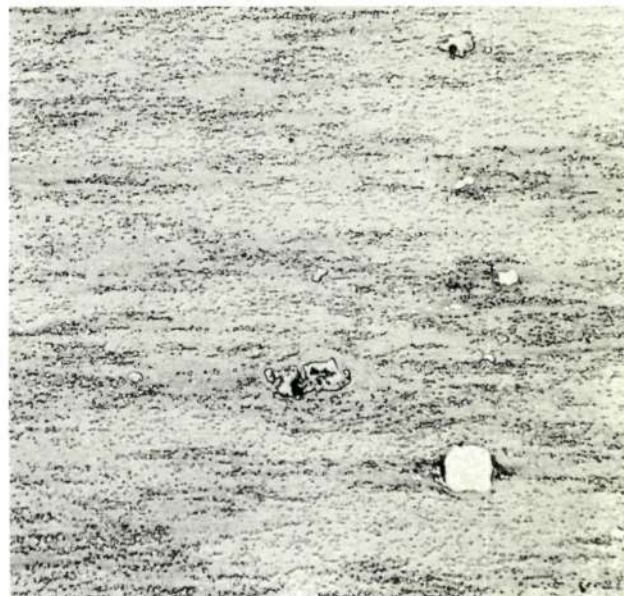
a - X-ray diffraction

b - wet analysis

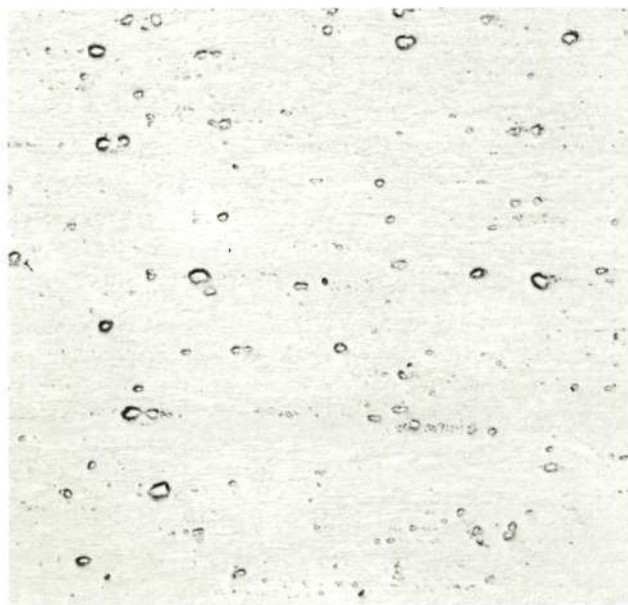
c - spectrographic analysis



a) Base Alloy



b) Base + Cr_3C_2



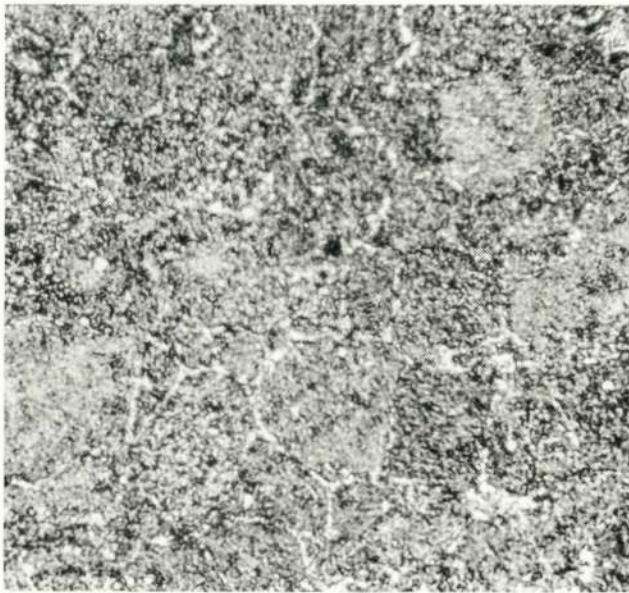
c) Base + TiC



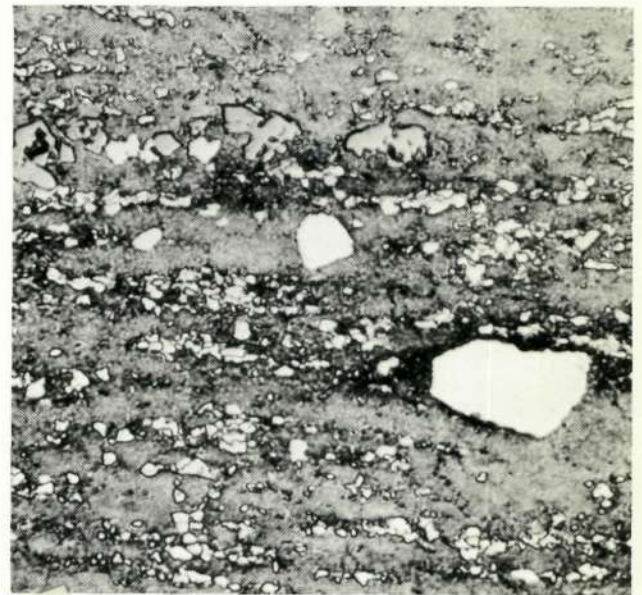
d) Base + VC

Figure 3. Light photomicrographs of MAR-M246 type superalloy powder made with various carbide additions (0.2 w/o C) and extruded at 2175°F (1190°C), 16:1 reduction ratio. All magnifications are 250X. All are longitudinal sections.

This page is reproduced at the back of the report by a different reproduction method to provide better detail.



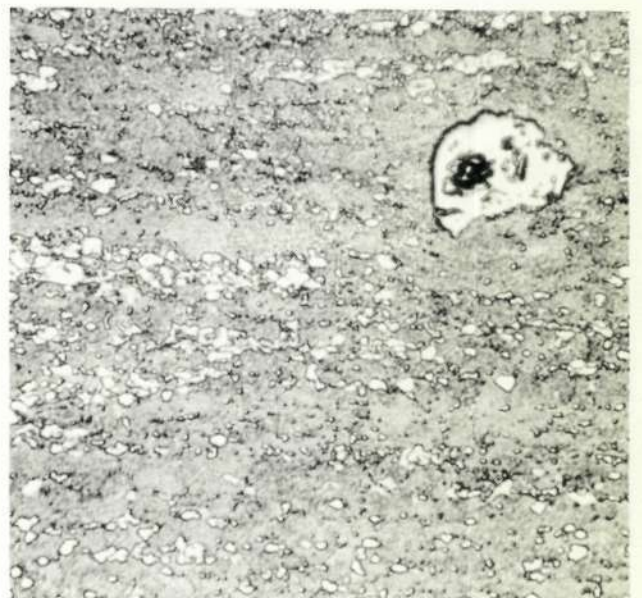
a) Base Alloy



b) Base + Cr_3C_2



c) Base + TiC



d) Base + VC

Figure 4. Light photomicrographs of MAR-M246 type superalloy powder made with various carbide additions (0.2 w/o C) and extruded at 2175°F (1190°C), 16:1 reduction ratio. All magnifications are 1000X. All are longitudinal sections.

This page is reproduced at the back of the report by a different reproduction method to provide better detail.

No recrystallization was observed in the material containing Cr_3C_2 . A considerable amount of banding was noted in the microstructure and was attributed to the large amounts of small precipitates (~2 microns) seen throughout the matrix. Numerous large (~24 microns) particles were also seen which appeared to be similar to those smaller precipitates responsible for banding. Microprobe analysis of these larger particles conducted later in the program indicated that they were MC type carbides. These results will be discussed in greater detail in the property optimization portion of the program. Although the manufacturer indicated that the Cr_3C_2 carbides were approximately 6-8 microns in size, numerous carbides observed in the microstructure were measured to be approximately 24 microns in size, indicating that some agglomeration occurred during handling, blending, and consolidation.

Recrystallization to an ASTM No. 7 grain size occurred in the alloy containing TiC, but only in the central portion of the extrusion. There was a moderate amount of banding throughout the microstructure, but the large particles found in extrusion #2 were not observed. The original carbides also appeared to be somewhat agglomerated as a result of the consolidation process.

Recrystallization to an ASTM No. 7 grain size was observed in the material containing VC but only in the central portion of the extrusion. There was a considerable amount of banding throughout the microstructure and the original carbides also appeared to be somewhat agglomerated.

In summary, metallographic examination revealed that the hot extrusion consolidation process produced completely dense bar stock, and that the original carbides had agglomerated. Considerable banding was observed throughout the microstructure and recrystallization took place in all extrusions except that to which Cr_3C_2 had been added.

3. As-Extruded Mechanical Properties

The tensile and stress rupture properties for these four alloys in the as-extruded condition are listed in Table 3 along with those for cast MAR-M246 for comparison purposes. The ultimate tensile strength and the 0.2 percent offset yield strength of all four powder alloys at room temperature and 1400°F (760°C) exceeded that of the cast alloy. This can be attributed to the extremely fine grain size (ASTM 7-8) of the extruded powder. No 1900°F (1038°C) tensile data were available for cast MAR-M246, but an extrapolation of the 1800°F (980°C) properties to 1900°F (1038°C) indicated that the powder product would not be as strong as the cast material because of the fine grain size. The best 1400°F (760°C) and 1900°F (1038°C) ultimate and 0.2 percent offset yield strength values of the powder product were exhibited by material containing TiC.

Table 3

Mechanical Properties for As-Extruded Material of First Alloy SeriesTensile Properties

Extrusion No.	Alloy	%C	Temperature		Ultimate Strength		0.2% Offset Yield Strength		R.A. %	Elongation %
			(°F)	(°C)	ksi	MN/m ²	ksi	MN/m ²		
1	Base	0.01	Room	22	235.9	1627	171.2	1180	15.7	14.2
			1400	760	181.4	1250	168.2	1160	2.0	3.1
			1900	1040	6.1	41.4	4.8	30.6	90.3	229.5
2	Base + Cr ₃ C ₂	0.21	Room	22	218.5	1507	171.0	1179	11.6	9.7
			1400	760	184.4	1271	168.7	1159	5.1	5.9
			1900	1040	6.1	41.4	3.0	20.7	97.3	552.9(3)
3	Base + TiC	0.21	Room	22	227.9	1572	171.7	1184	13.4	12.7
			1400	760	195.5	1348	174.6	1204	5.5	4.3
			1900	1040	42.9	296	18.3	126	10.8	12.4
4	Base + VC	0.19	Room	22	227.1	1567	169.3	1167	11.7	11.1
			1400	760	184.2	1270	170.4	1175	4.7	4.6
			1900	1040	41.6	286	12.4	85.7	16.4	18.6
	Cast MAR-M246(1)	0.15	Room	22	140.0	965	125.0	862	(2)	5.0
			1400	760	150.0	1034	125.0	862	-	5.0
			1800	980	80.0	552	55.0	379	-	8.0
			1900(4)	1040	62.5	431	37.0	255	-	-

Table 3 (continued)

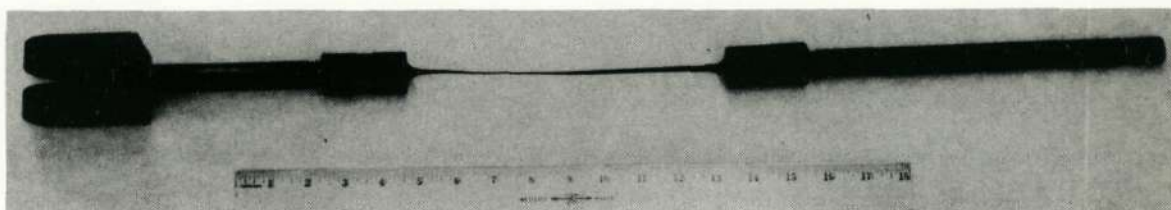
1900°F (1040°C)/15 ksi (104 MN/m²) Stress Rupture Properties

<u>Extrusion No.</u>	<u>Alloy</u>	<u>%C</u>	<u>Rupture Life (Hours)</u>	<u>% Elongation</u>	<u>% Reduction in Area</u>
1	Base	0.01	0.6 0.4	12.7 14.8	15.7 17.1
2	Base + Cr ₃ C ₂	0.21	0.1 0.2	132.0 65.1	67.0 54.5
3	Base + TiC	0.21	1.1 1.0	25.8 26.6	32.6 31.1
4	Base + VC	0.19	0.5 0.5	38.9 40.1	40.9 43.9
	MAR-M246(1) Alloy with carbon	0.15	100 hours at 20,000 psi (138 MN/m ²)		

- (1) Data from International Nickel Data Brochure, 1964 Edition.
 (2) Data not available.
 (3) Test was concluded when the specimen extended out of the furnace heat zone.
 (4) Extrapolated data.

The percent elongation for all four powder alloys at room temperature exceeded that of the cast alloy. Values were slightly lower at 1400°F (760°C) with the exception of material containing Cr₃C₂, but this was attributed to the fact that no aging treatments were applied to promote a desirable discrete carbide particle precipitation in the grain boundaries. This projection of the 1800°F (980°C) tensile data to 1900°F (1038°C) for cast MAR-M246 also indicated that the elongation of the four powder alloys would be superior at 1900°F (1038°C). Both the base powder and the alloy containing Cr₃C₂ evidenced superplasticity at 1900°F (1038°C). Failure occurred in the base material after an elongation of 229.5 percent, while failure did not occur in the Cr₃C₂ alloy even after 552.9 percent elongation. (This test was concluded when the specimen extended beyond the furnace heat zones, Figure 5.) These superplasticity results indicated that there is considerable potential for working these materials into part configurations.

The as-extruded stress rupture results at 1900°F (1038°C) were quite poor in relation to those of the as-cast material, Table 3. Although the alloys showed appreciable ductility, the rupture lives were low. The extremely fine grain size of the as-extruded bar stock was responsible for these low rupture life values.



NOT REPRODUCIBLE

Figure 5. 1900°F (1040°C) tensile specimen of alloy containing Cr_3C_2 at 0.21 w/o C. Test halted at 552.9% elongation when specimen extended out of furnace heat zone.

V TASK II - PROPERTY OPTIMIZATION

A. Series 1 - Selection of Most Desirable Metastable Carbide

1. Materials and Procedures

a. Heat Treatment Studies

The high temperature heat treatment studies were conducted under a protective atmosphere of argon and vacuum. Argon heat treatments were conducted in a Lindberg Hevi-Duty tube furnace equipped with a 0.25 inch (6.4 mm) thick alumina tube. Vacuum heat treatments were conducted in a Vacuum Industries, Inc. Model 6615-2100 unit with a vacuum capability of 3.0×10^{-7} mm (4 x 10⁻⁵ N/m²). All heat treatments were evaluated according to resultant ASTM grain sizes utilizing the metallographic preparation procedures described previously.

b. Mechanical Property Evaluations

Mechanical property tests were performed on material after heat treatments which resulted in an ASTM No. 2-3 grain size and a discrete particle carbide distribution in the grain boundary. While the high temperature solution heat treatments varied for each alloy, the aging treatments were identically conducted in air and consisted of 2000°F (1090°C)/4 hours/air cool, 1600°F (870°C)/8 hours/air cool, 1800°F (980°C)/4 hours/air cool, 1200°F (650°C)/24 hours/air cool, and 1400°F (760°C)/8 hours cool. All testing was done according to procedures described previously in Task I.

As-extruded material in the form of completely decanned 3 inch (7.65 cm) long by 0.5 inch (1.27 cm) diameter bars were sent to the NASA Program Manager for his evaluation including stress rupture tests at 1200°F (650°C)/150 ksi (1050 MN/m²) and 1400°F (760°C)/62 ksi (430 MN/m²), and were sent back to TRW for metallographic evaluation.

c. Vacuum Wash Procedural Change

During the preparation of specimens for mechanical property tests, a thermocouple malfunction in the vacuum furnace caused all the base alloy specimens to melt and another 8 pound (3.6 kg) extrusion of this alloy was made. This procedure was identical to that for the first four extrusions except that vacuum washing techniques were employed in an attempt to reduce the gas porosity resulting from the high temperature solution treatments used in the heat treatment study. During pump-down in the electron beam welding chamber, as shown schematically in Figure 6, induction coils were placed around the extrusion can which was then heated to 1000°F (540°C) overnight, while a vacuum of 1×10^{-5} mm (1.33 x 10⁻³ N/m²) was maintained.

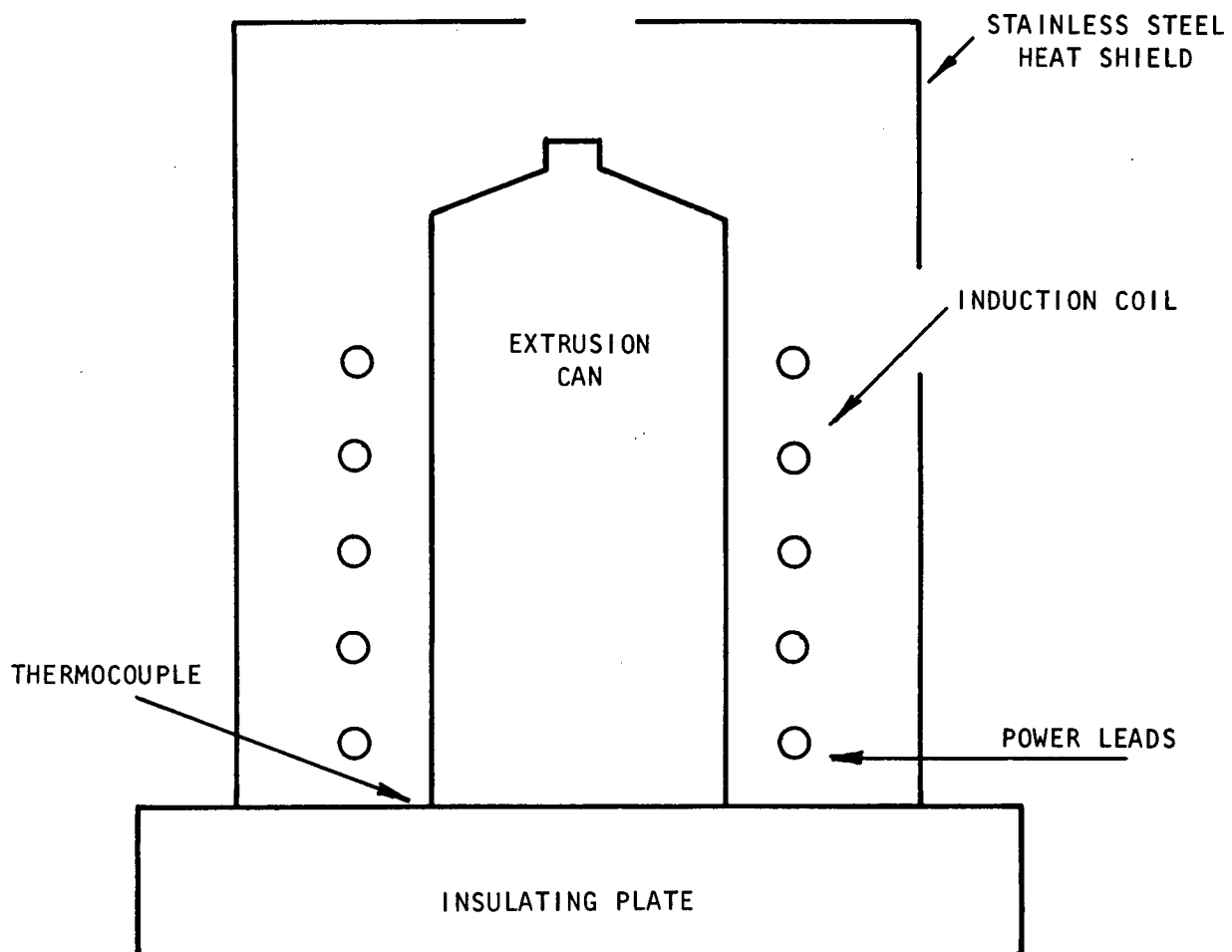


Figure 6. Schematic illustration of apparatus inside electron beam welding chamber for vacuum washing extrusion billets.

After sealing, the billet was single extruded at 2175°F (1190°C) at a 16:1 reduction and ram speed of 1.7 inches (4.3 cm)/second with a resultant breakthrough pressure of 82.8 ksi (572.3 MN/m²). Radiographic and light metallographic analyses were conducted similar to that for the first four extrusions. Heat treatment studies to produce an ASTM 2-3 grain size and mechanical property evaluations were also conducted.

2. Results and Discussion

a. Argon Atmosphere Solution Heat Treatments

i. Single Exposure Solution Heat Treatments

The initial attempts to produce an ASTM No. 2-3 grain size in the first four extrusions consisted of 6 hour solution heat treatments in an argon protective atmosphere starting at 2250°F (1232°C). The grain size versus solution temperature for the various alloys are shown in Figure 7. All alloys evidenced some grain growth, but in all instances the major part of this growth occurred after the initiation of incipient melting. The base alloy experienced the greatest grain growth followed by the alloy containing TiC. The lowest temperature for incipient melting was recorded by the base alloy.

The light photomicrograph of Figure 8a shows an example of the microstructure at incipient melting for the base alloy plus Cr₃C₂. In addition to grain size and incipient melting, several other features were noted from the metallographic analysis of these solution heat treated specimens. Comparison of this microstructure with that of Figure 3b indicates that many of the small precipitates observed in the as-extruded microstructure were dissolved during the high temperature exposure. Also, as shown in Figure 8b for the base alloy plus VC, the original blended carbides started to decompose at temperatures below the incipient melting point which resulted in the formation of an angular phase later identified as an MC carbide.

These results indicated that some grain growth was achieved in these alloys but only after the occurrence of incipient melting. Examination of the microstructures revealed that incipient melting should be avoided for two reasons. First, small areas of microporosity appear, especially at grain boundary triple points which may be deleterious to high temperature properties, Figures 9a and 9b. Second, continuous microconstituents, formed within the resolidified material, Figure 9c, could be detrimental to the high temperature properties.

In an attempt to promote the desired grain size in the microstructure but avoid incipient melting, a number of single exposure solution heat treatments were investigated which involved either longer exposure times at temperatures slightly below or shorter exposure times at temperatures slightly above the incipient melting points. The results of these solution treatments are listed in Table 4 and are compared to the treatments causing

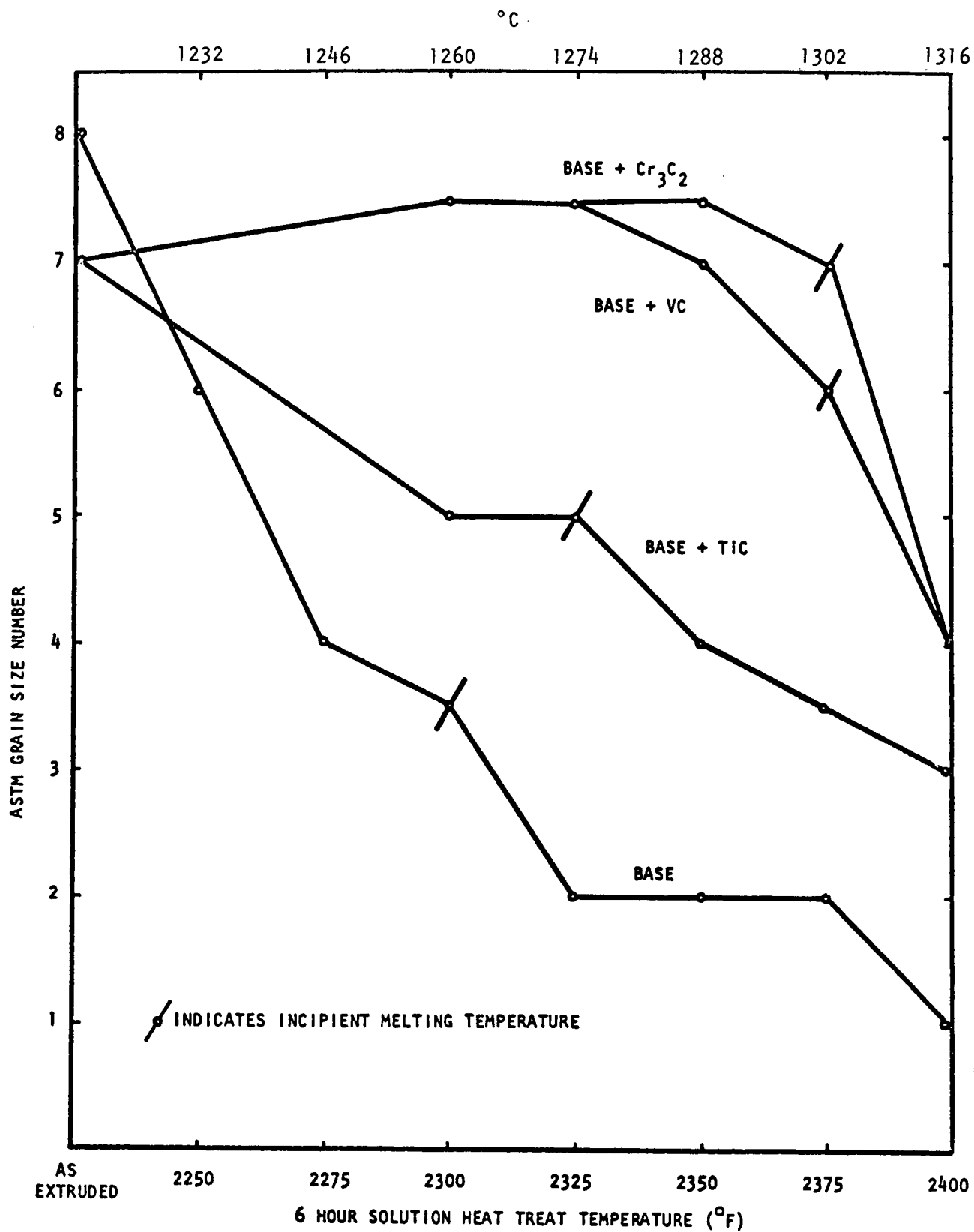
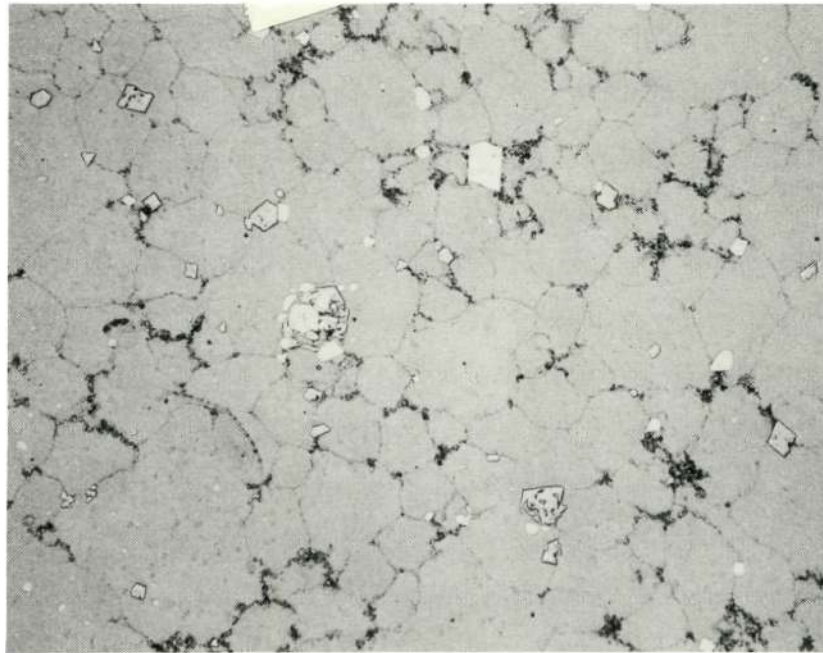
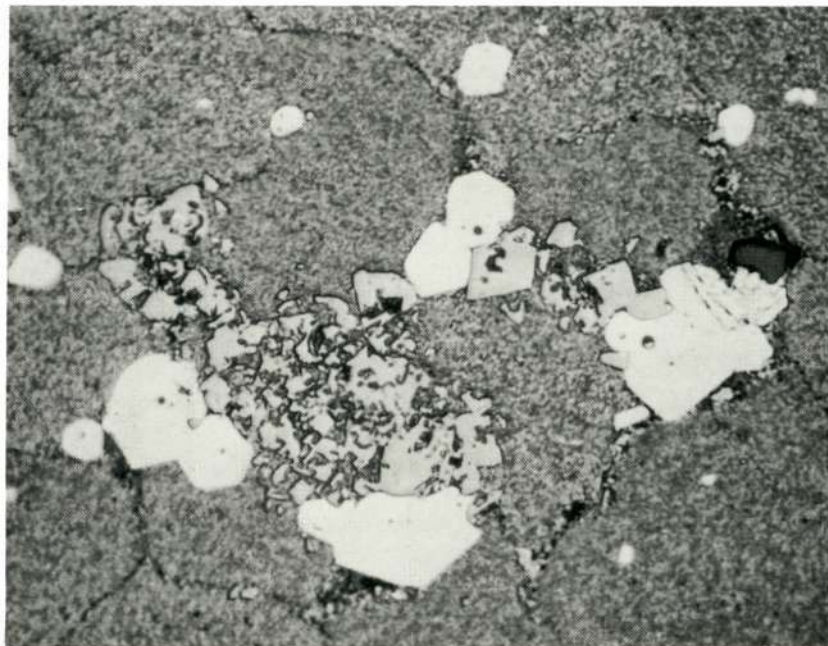


Figure 7. Grain size versus solution temperature (6 hour exposure) for extruded superalloy powder of modified MAR-M246 composition.



a) Base alloy + Cr_3C_2 , 2375°F (1300°C)
ASTM 7 (35 microns) 250X, etched.



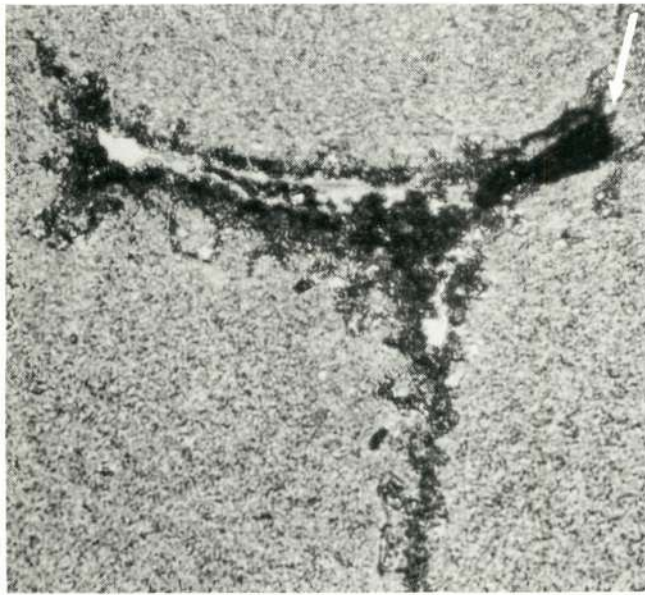
b) Base alloy + VC, 2375°F (1300°C)
500X, etched.

Figure 8. Light photomicrographs of modified MAR-M246 superalloy powder extrusions at 0.2 w/o C after 6 hour solution heat treatments in argon showing incipient melting a) and decomposition of original carbides, b) all are transverse sections.

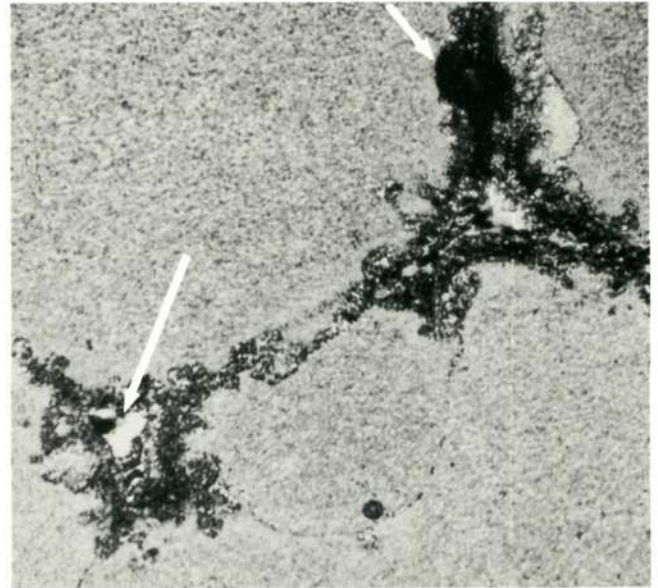
This page is reproduced at the back of the report by a different reproduction method to provide better detail.

Grain Boundary

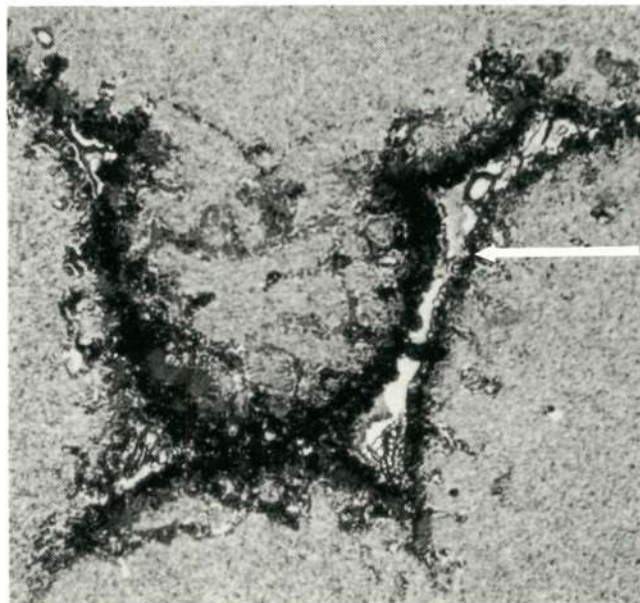
Triple Point Microporosity



a) Base alloy, 2300°F (1260°C)



b) Base alloy, 2300°F (1260°C)



Continuous Resolidified
Material

c) Base alloy, 2325°F (1270°C)

This page is reproduced at the back of the report by a different reproduction method to provide better detail.

Figure 9. Light photomicrographs of modified MAR-M246 superalloy extrusions after 6 hour solution heat treatments in argon. Note the potentially harmful features of incipient melting at the grain boundary triple points. Transverse sections, 1000X magnification, etched.

Table 4

Results of Single Exposure Solution Heat Treatments

Extrusion No.	Alloy	Incipient Melting Temperature		Exposure Time (Hours)	ASTM Grain Size No.	Remarks
		°F	°C			
1	Base	2275	1246	6	3.5	Incipient Melting
		2300	1260	24	3.5	No Melting
		2325	1274	4	3	Incipient Melting
2	Base + Cr ₃ C ₂	2375	1300	6	7	Incipient Melting
		2400	1315	3	5	Incipient Melting
		2425	1330	1	3.5	Incipient Melting
3	Base + TiC	2325	1274	6	5	Incipient Melting
		2350	1288	4	4.5	Incipient Melting
		2375	1300	2	4	Incipient Melting
		2400	1315	1	4	Incipient Melting
4	Base + VC	2375	1300	6	6	Incipient Melting
		2400	1315	3	4.5	Incipient Melting
		2425	1330	1	4	Incipient Melting

incipient melting after 6 hours exposure. Although grain growth occurred in shorter times at temperatures above that which caused incipient melting after 6 hours, the shorter exposures still produced appreciable amounts of incipient melting at the grain boundary triple points. In addition to this, the size of the grains was still below the program requirement of ASTM 2-3.

In order to determine what stress rupture life could be expected with the grain size developed from these solution treatments and what, if any, were the detrimental effects of utilizing a structure containing incipient melting, stress rupture tests at 1900°F (1038°C)/15 ksi (104 MN/m²) were conducted on material receiving several different heat treatments. The base alloy and the base alloy plus TiC were selected for these tests and three conditions were evaluated for each alloy: (1) solution heat treated 6 hours at 25°F (15°C) below the point of incipient melting, (2) solution heat treated 6 hours at 25°F (15°C) below the point of incipient melting plus the five-step aging treatment, and (3) solution heat treated 6 hours at 25°F (15°C) above incipient melting plus the five-step aging treatment. Incipient melting occurred at 2300°F (1260°C) for the base alloy and at 2325°F (1270°C) for the base alloy plus TiC. The results are listed in Table 5.

Comparison with the as-extruded stress rupture results listed in Table 3 indicated that heat treatment improved the rupture life. However, in all cases the rupture lives were substantially below that of as-cast MAR-M246. Metallographic examination of the failed bars revealed that fracture paths were intergranular and in many instances cracks initiated at areas of porosity within the microstructure, Figure 10. This would indicate that porosity obscured the effects of the variables of interest, grain size and incipient melting. It is significant to note that this porosity was not evident in the as-extruded microstructure, but appeared only after the application of high temperature heat treatments. It was also noted that the grain sizes obtained in the base alloy specimens were slightly smaller than expected for these same temperatures, while those for the base alloy plus TiC were about as expected, Figure 7.

Subsequent to these stress rupture tests, metallographic specimens used in the 6 hour solution heat treat study were re-examined semi-quantitatively for amounts of porosity and incipient melting. Three major categories were designated for the visual porosity measurements, 0.2%, 0.5% and 1.0%. The system employed to determine the amounts of incipient melting in a microstructure involved recording the extent to which a particular grain was surrounded by resolidified film. The following tabulation was used:

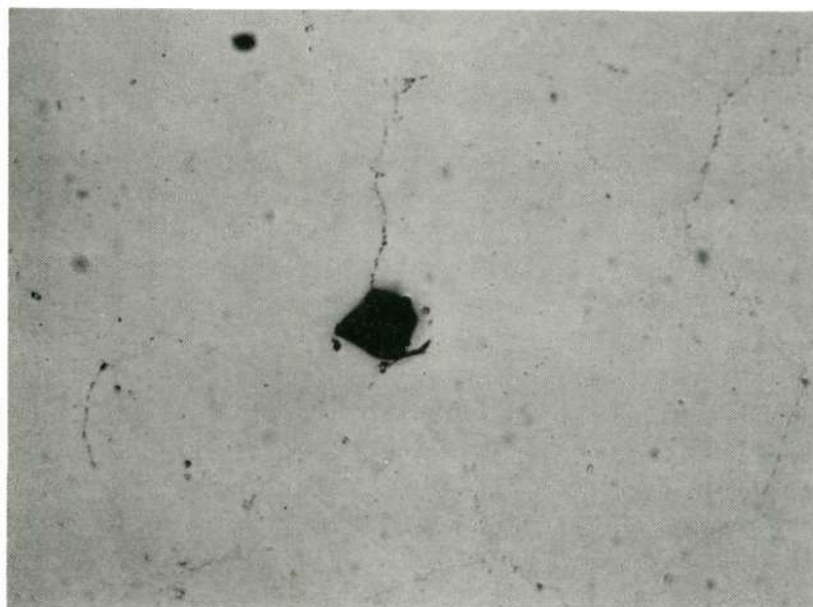
- 100% - all dendritic structure indicating a completely cast microstructure.
- 40% - film surrounding entire grains
- 25% - film 3/4 surrounding grains

Table 5
1900°F (1040°C)/15 ksi (104 MN/m²) Stress Rupture Properties
for Extruded and Heat Treated First Series Alloys,
Base and Base + TiC

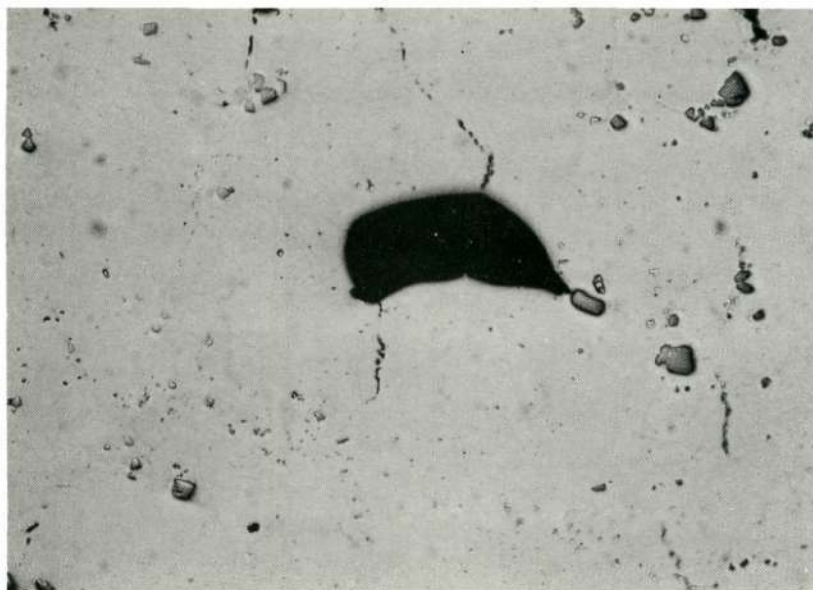
Extrusion No.	Alloy	Heat Treatment	Rupture Life (hours)	R.A. %	Elongation %	ASTM Grain Size
1	Base	6 hours @ 2275°F (1246°C)	1.2	1.2	2.0	5
1	Base	6 hours @ 2275°F (1246°C), Full age*	0.9	0.8	2.2	6
1	Base	6 hours @ 2325°F (1274°C), Full age	1.5	0	1.5	3
3	Base + TiC	6 hours @ 2300°F (1260°C)	5.2	1.6	1.7	4
3	Base + TiC	6 hours @ 2300°F (1260°C), Full age	3.9	1.2	2.2	5
3	Base + TiC	6 hours @ 2350°F (1288°C), Full Age	3.0	1.6	1.6	4
	Cast MAR-M246**	-	100 hours at 20 ksi (138 MN/m ²)			

* Full age: 4 hours @ 2000°F (1093°C)/8 hours @ 1600°F (870°C)/4 hours
@ 1800°F (980°C)/24 hours @ 1200°F (650°C)/8 hours @ 1400°F (760°C).

** Data from International Nickel Data Brochure, 1964 Edition.



a) Base alloy, 6 hours at 2325°F (1270°C)
+ full age, 1.5 hours 0% R.A., 1.5%
elongation, 250X, unetched.



b) Base + TiC, 6 hours at 2350°F (1290°C)
+ full age, 5.2 hours, 1.6% R.A., 1.7%
elongation, 250X, unetched.

This page is reproduced at the
back of the report by a different
reproduction method to provide
better detail.

Figure 10. Light photomicrographs of first series base alloy and base alloy + TiC after 1900°F (1040°C)/15 ksi (104 Mn/m²) stress rupture tests showing cracks initiated by porosity. All are longitudinal sections. Full age as described in Table 5.

20% - film 1/2 surrounding grains

10% - triple point melting only

0% - no evidence of incipient melting

The results are illustrated graphically in Figures 11 through 14. As temperature increased, both the amount of porosity and incipient melting increased. Comparison of these plots with those of Figure 7 for grain size indicated that although grain size increased with temperature, the amount of porosity and incipient melting also increased to a substantial degree.

ii. Multiple Exposure (Homogenization) Solution Heat Treatments

The metallographic evaluation of specimens after single solution heat treatments indicated that some grain growth was achieved in the powder extrusions, but this was accompanied by an undesirable amount of incipient melting and porosity. As indicated by the results of Figures 11-14, porosity was present even in the absence of incipient melting. In order to increase the grain size without the accompanying incipient melting, solution heat treatments were applied, the first stages of which were conducted below the incipient melting temperature in order to homogenize the grain boundary areas. Successive exposures were then applied at temperatures above the original incipient melting point. The metallographic analysis results of these multiple solution heat treatments are shown in Table 6.

The most significant conclusion reached from these studies was that at temperatures above 2275°F (1246°C) porosity was present in the microstructure. As shown in Figure 15a, this porosity was not associated with the zone of incipient melting at the grain boundaries, but rather, was located throughout the grains. Semi-quantitative metallographic analysis revealed that at the lowest levels, approximately 0.2% of the structure consisted of voids, and that this percentage increased with increasing temperature and time at temperature. The highest percentage, 1.0% was recorded in specimens heat treated at or near 2400°F (1316°C). As shown by the stress rupture results, this amount of porosity would be highly detrimental to high temperature properties.

For each of the four alloys studied, at least one heat treatment was developed which was successful in promoting increased grain growth and a reduction in the amount of incipient melting over that experienced with the earlier 6 hour solution treatments. However beneficial these results were, the presence of porosity in the structure would still be the dominant factor as far as high temperature properties were concerned and its removal was imperative.

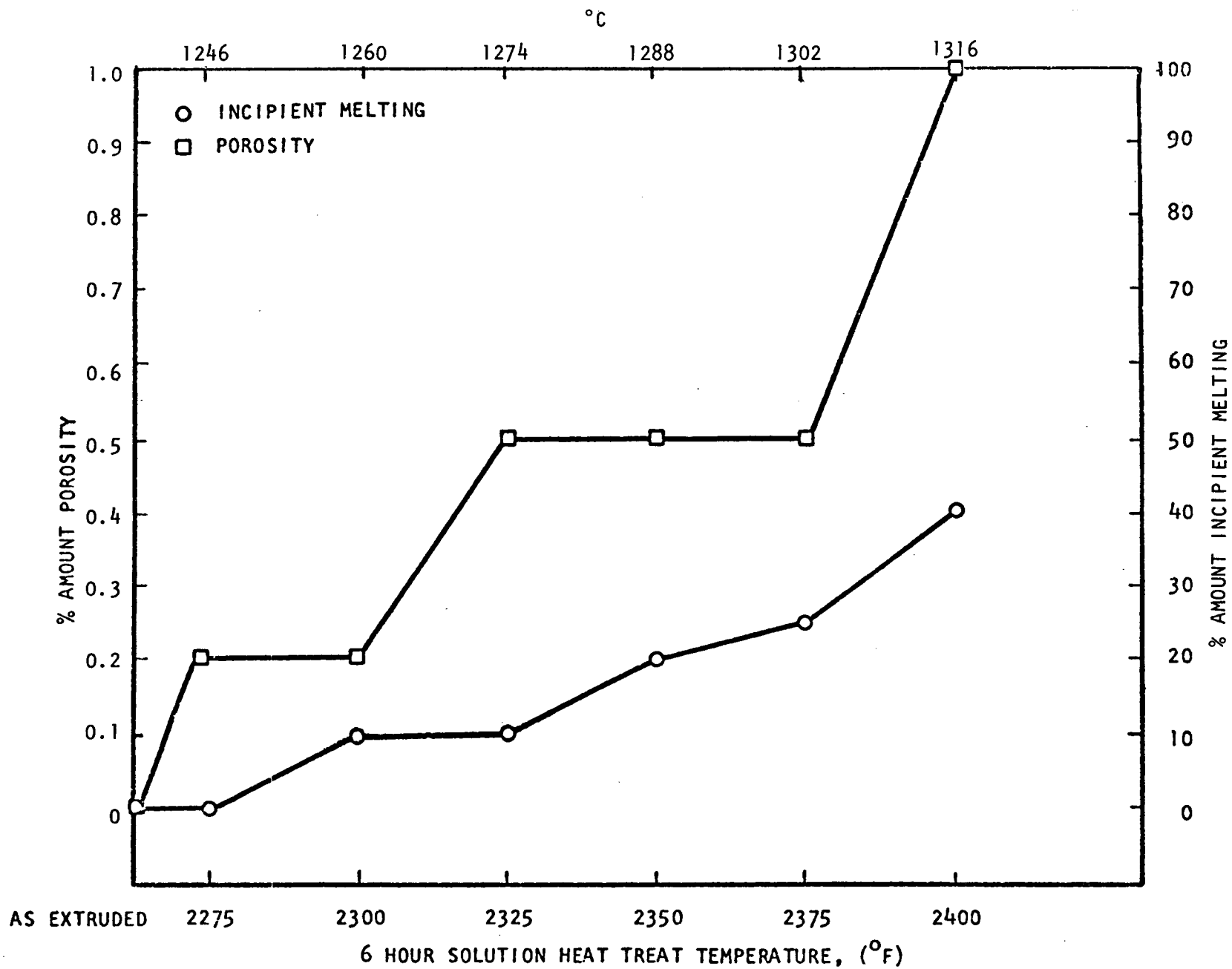


Figure 11. Amount incipient melting and porosity for base alloy for material solution heat treated 6 hours in argon.

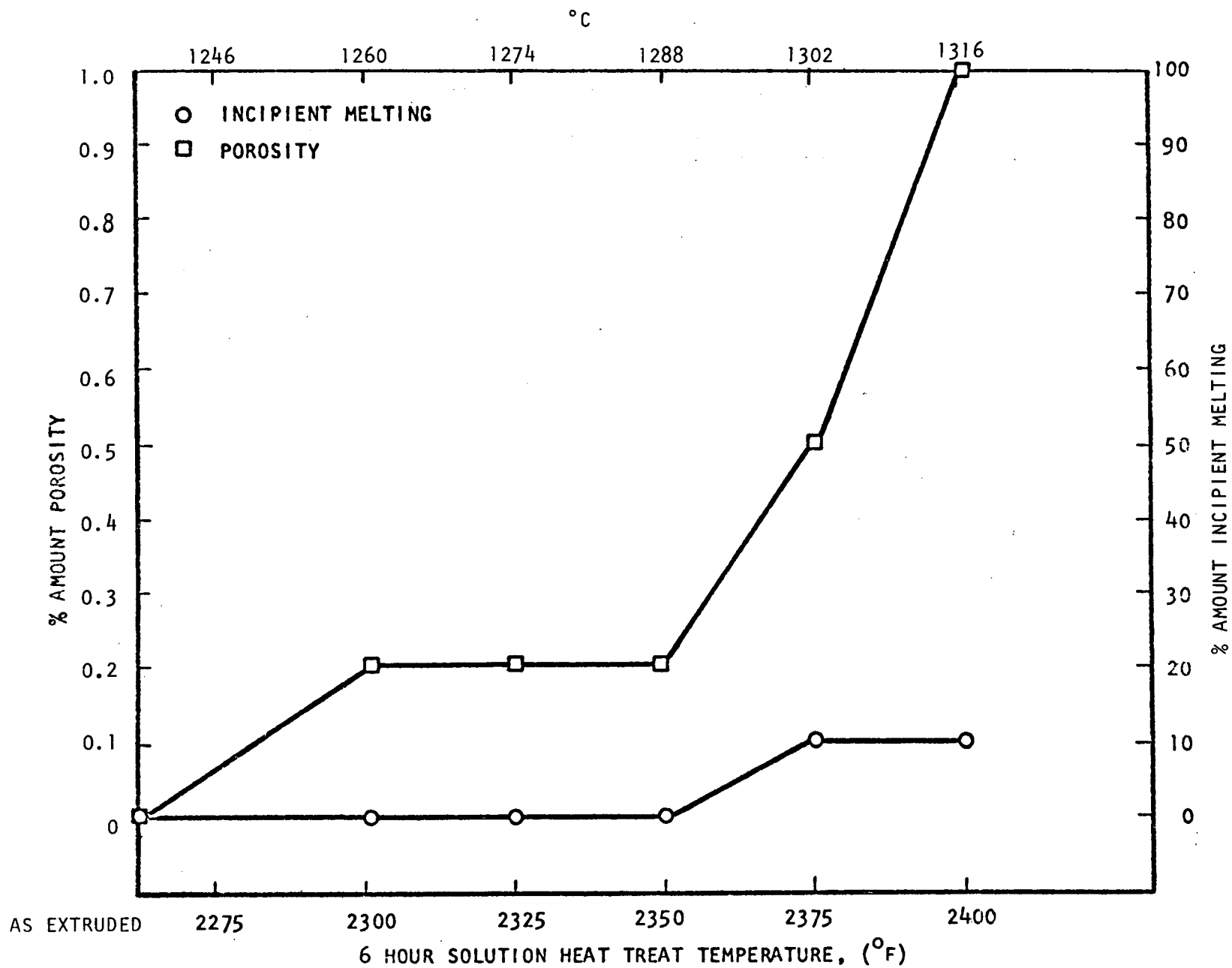


Figure 12. Amount incipient melting and porosity for base alloy + Cr_3C_2 for material solution heat treated 6 hours in argon.

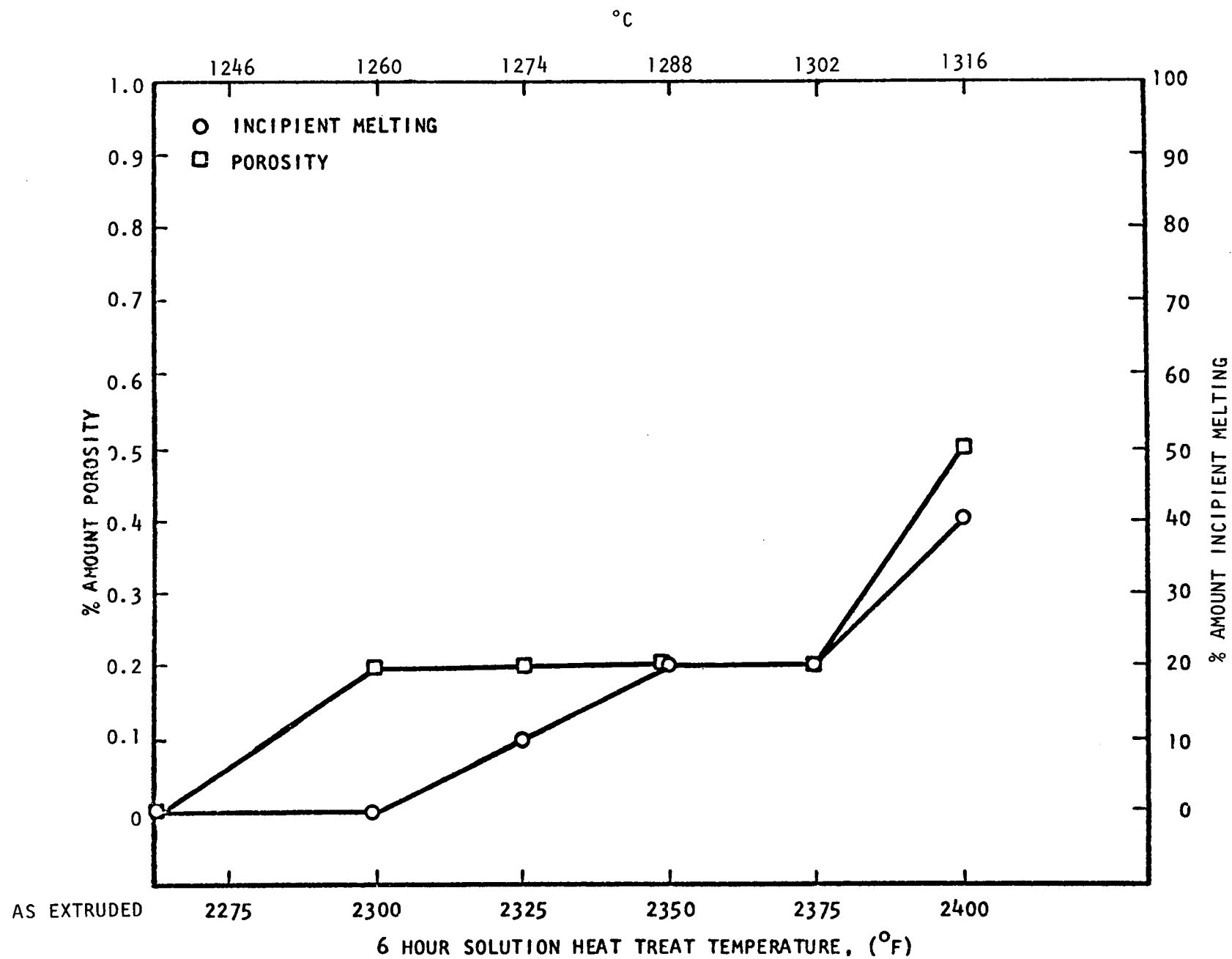


Figure 13. Amount incipient melting and porosity for base alloy + TiC for material solution treated 6 hours in argon.

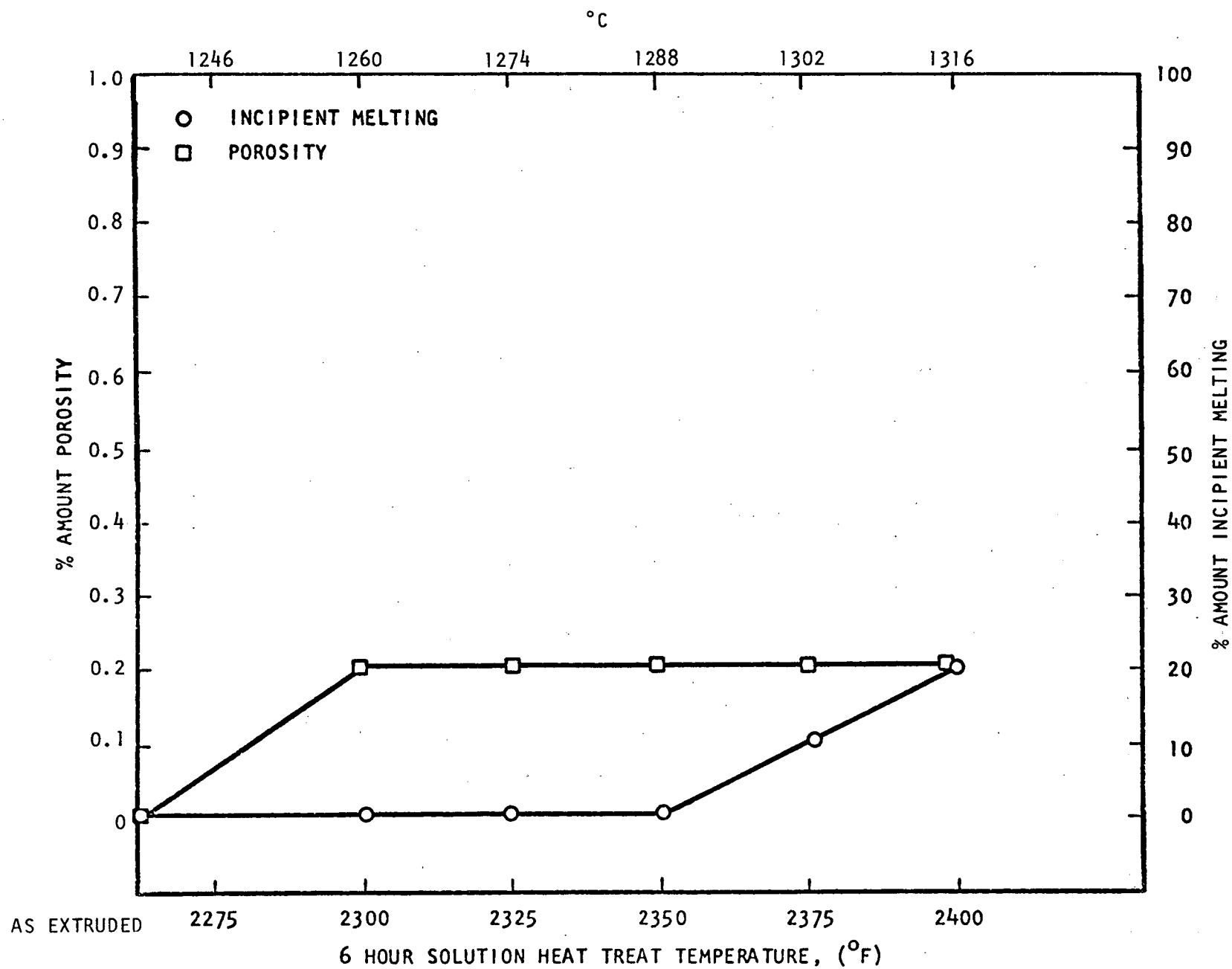


Figure 14. Amount incipient melting and porosity for base alloy + VC for material solution treated 6 hours in argon.

Table 6

Metallographic Results of Multiple Solution Heat Treatments in Argon
Applied to the Four Alloys of Series I

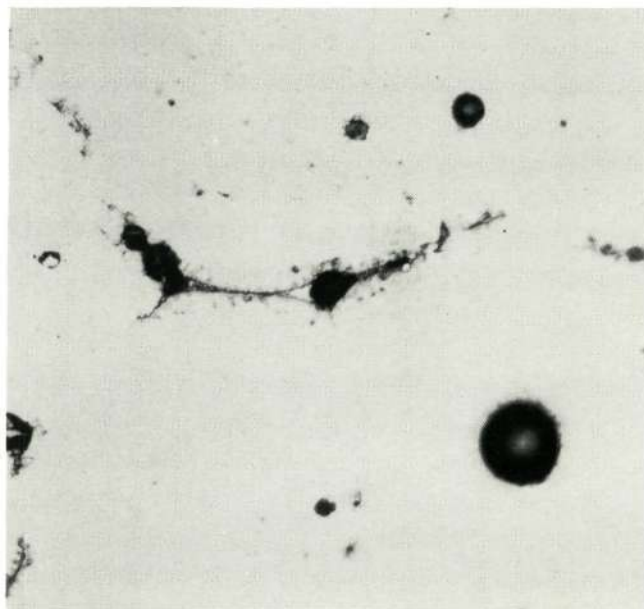
<u>Alloy</u>	<u>Heat Treatment</u>	<u>ASTM Grain Size No.</u>	<u>Porosity % (1)</u>	<u>Incipient Melting % (1)</u>
Base	6 hours @ 2300°F (1260°C) (2)	3.5	0.2	10
	24 hours @ 2275°F (1246°C)	1.5	0.5	20
	4 hours @ 2325°F (1274°C)			
	24 hours @ 2275°F (1246°C)	2	0.5	10
	2 hours @ 2375°F (1301°C)			
Base + Cr ₃ C ₂	6 hours @ 2375°F (1301°C) (2)	7	0.5	10
	24 hours @ 2350°F (1288°C)	2.5	1.0	25
	3 hours @ 2400°F (1315°C)			
	24 hours @ 2350°F (1288°C)	3	1.0	40
	2 hours @ 2425°F (1330°C)			
	24 hours @ 2350°F (1288°C)	2	1.0	25
	2 hours @ 2400°F (1315°C)			
	1 hour @ 2425°F (1330°C)			
	6 hours @ 2350°F (1288°C)	4	0.5	100
	3 hours @ 2400°F (1315°C)			
	2 hours @ 2425°F (1330°C)			
	1 hour @ 2450°F (1343°C)			
Base + TiC	6 hours @ 2325°F (1274°C) (2)	5	0.2	10
	24 hours @ 2300°F (1260°C)	3.5	0.2	0
	3 hours @ 2375°F (1301°C)			
	24 hours @ 2300°F (1260°C)	3.5	0.5	10
	1 hour @ 2400°F (1315°C)			
	24 hours @ 2300°F (1260°C)	4.5	0.2	10
	3 hours @ 2350°F (1288°C)			
	2 hours @ 2375°F (1301°C)			
	6 hours @ 2300°F (1260°C)	4.5	0.5	0
	4 hours @ 2325°F (1274°C)			
	6 hours @ 2300°F (1260°C)	3	1.0	0
	4 hours @ 2325°F (1274°C)			
	3 hours @ 2350°F (1288°C)			
	2 hours @ 2375°F (1301°C)			

Table 6 (continued)

<u>Alloy</u>	<u>Heat Treatment</u>	<u>ASTM Grain Size No.</u>	<u>Porosity % (1)</u>	<u>Incipient Melting % (1)</u>
Base + TiC	6 hours @ 2300°F (1260°C)	2	1.0	25
	4 hours @ 2325°F (1274°C)			
	3 hours @ 2350°F (1288°C)			
	2 hours @ 2375°F (1301°C)			
	1 hour @ 2400°F (1315°C)			
Base + VC	6 hours @ 2375°F (1302°C) (2)	6	0.2	10
	24 hours @ 2350°F (1288°C)	2.5	0.5	40
	3 hours @ 2400°F (1315°C)			
	24 hours @ 2350°F (1288°C)	2	1.0	40
	2 hours @ 2425°F (1330°C)			
	24 hours @ 2350°F (1288°C)	2	1.0	40
	2 hours @ 2400°F (1315°C)			
	1 hour @ 2425°F (1330°C)			
	6 hours @ 2350°F (1288°C)	4	0.5	100
	3 hours @ 2400°F (1315°C)			
	2 hours @ 2425°F (1330°C)			
	1 hour @ 2450°F (1343°C)			

(1) Rating system described in text.

(2) Result from initial 6 hour solution heat treatment, Figure 7.



a) Base alloy, 24 hours at 2275°F (1250°C) argon, 4 hours at 2325°F (1270°C) argon, 250X, unetched, transverse. Porosity within grains.



b) Base Alloy + VC, 1 hour at 2425°F (1330°C) argon, 250X etched, transverse. Incomplete decomposition of original carbide.



c) Base + TiC, 6 hours at 2350°F (1290°C) argon + full age, 5.2 hours, 1.6% R.A. 1.7% elongation, 500X, etched, longitudinal. Discrete particle grain boundary precipitation.

This page is reproduced at the back of the report by a different reproduction method to provide better detail.

Figure 15. Light photomicrographs of extruded powder of first series alloys showing a) porosity within grains, b) incomplete decomposition of original carbide, c) discrete particle grain boundary precipitation. Full age as described in Table 5.

While grain growth to an ASTM No. 2-3 grain size was the primary objective of these heat treatments, the decomposition of the original blended carbides was also observed. It was noted that even in specimens where extensive amounts of incipient melting occurred, Figure 15b, the original carbides had not decomposed completely. As shown in the microstructure of one of the stress rupture specimens, Figure 15c, complete decomposition of the original carbides was not necessary for discrete particle precipitation to occur in the grain boundary regions.

b. Solution Heat Treatments in Vacuum

In order to increase the grain size in the extruded alloys but avoid excessive amounts of porosity, a series of vacuum heat treatments were investigated to promote diffusion of the internal gasses out of the microstructure. These treatments included multiple exposures starting below incipient melting to produce the proper grain size. The metallographic analysis results for these heat treatments are listed in Table 7. Exposures in vacuum at 2250°F (1232°C) produced a substantial decrease in the amount of porosity observed in the microstructure, but exposures at 2350°F (1288°C) or above still were accompanied by considerable porosity. In addition to this, grain size was also observed to increase substantially at 2250°F (1232°C) for the base alloy, the base alloy plus TiC, and for the base alloy plus VC. As a result of these vacuum heat treatments, a grain growth treatment was selected for each alloy and is listed in Table 8.

For the base alloy the 12 hour exposure at 2250°F (1232°C) produced an ASTM No. 2 grain size, satisfactory for stress rupture testing at 1900°F (1038°C). In addition, the porosity level was quite low. As indicated in Table 8, the heat treatment for the base alloy plus Cr₃C₂ produced an ASTM grain size of only 6.5. It was believed, however, that to heat treat at higher temperatures in order to increase this grain size would result in an unacceptable amount of porosity. For the base alloy plus VC, a series of thermal exposures consisting of 6 hours in vacuum at 2250°F (1232°C), 6 hours in vacuum at 2350°F (1288°C), and 2 hours in vacuum at 2400°F (1315°C) produced a microstructure consisting of large secondary recrystallized grains in a matrix of ASTM No. 7 grain size. The structure consisted of equiaxed grains of ASTM No. 2 throughout approximately 90% of the microstructure, with small clusters of ASTM No. 7 in the matrix.

c. Vacuum Washed Base Alloy

Radiographic analysis revealed no macroscopic defects throughout the extruded bar stock. Material in the as-extruded condition was fully dense and after the vacuum heat treatment exhibited the desired ASTM No. 2-3 grain size. As shown in Figure 16, the vacuum wash treatment prior to extrusion did not completely remove all the internal gases, but the porosity measurements were the lowest recorded for the first series alloys.

Table 7

Metallographic Results of Solution Heat Treatments in Vacuum
Applied to the Four Alloys of Series I

<u>Alloy</u>	<u>Heat Treatment</u>	<u>ASTM Grain Size No.</u>	<u>Porosity % (1)</u>	<u>Incipient Melting % (1)</u>
Base	6 hours @ 2300°F (1260°C) Argon (2)	3.5	0.2	10
	4 hours @ 2250°F (1232°C) Vacuum	2.5	0.2	0
	4 hours @ 2350°F (1288°C) Argon			
	24 hours @ 2275°F (1246°C) Vacuum	>1	0.5	0
	6 hours @ 2325°F (1274°C) Argon			
	24 hours @ 2250°F (1232°C) Vacuum	1	≤0.1	0
	12 hours @ 2250°F (1232°C) Vacuum	2	≤0.1	0
	24 hours @ 2250°F (1232°C) Vacuum	2	0.1	0
	4 hours @ 2350°F (1288°C) Vacuum			
Base + Cr ₃ C ₂	6 hours @ 2375°F (1301°C) Argon (2)	7	0.5	10
	4 hours @ 2250°F (1232°C) Vacuum	5.5	≤0.1	0
	4 hours @ 2350°F (1288°C) Argon			
	24 hours @ 2350°F (1288°C) Vacuum	4	1.0	0
	12 hours @ 2250°F (1232°C) Vacuum	7	<0.1	0
	4 hours @ 2350°F (1288°C) Vacuum			
	24 hours @ 2250°F (1232°C) Vacuum	6	<0.1	0
	4 hours @ 2350°F (1288°C) Vacuum			
	24 hours @ 2250°F (1232°C) Vacuum	5	1.0	0
	4 hours @ 2350°F (1288°C) Vacuum			
	4 hours @ 2400°F (1316°C) Vacuum			
	4 hours @ 2250°F (1232°C) Vacuum	8	<0.1	0
	4 hours @ 2350°F (1288°C) Vacuum			
	48 hours @ 2250°F (1232°C) Vacuum	8	<0.1	0
	72 hours @ 2250°F (1232°C) Vacuum	8	<0.1	0
	72 hours @ 2250°F (1232°C) Vacuum	6.5	0.1	0
	4 hours @ 2350°F (1288°C) Vacuum			
Base + TiC	6 hours @ 2325°F (1274°C) Argon (2)	5	0.2	10
	4 hours @ 2250°F (1232°C) Vacuum	4	≤0.1	0
	4 hours @ 2350°F (1288°C) Argon			
	24 hours @ 2275°F (1246°C) Vacuum	2.5	1.0	10
	24 hours @ 2300°F (1260°C) Argon			
	3 hours @ 2375°F (1300°C) Argon			

Table 7 (continued)

<u>Alloy</u>	<u>Heat Treatment</u>	<u>ASTM Grain Size No.</u>	<u>Porosity (1)</u>	<u>Incipient Melting (1)</u>
Base + TiC	24 hours @ 2275°F (1246°C) Vacuum	2	0.5	20
	6 hours @ 2300°F (1260°C) Argon			
	4 hours @ 2325°F (1274°C) Argon			
	3 hours @ 2350°F (1288°C) Argon			
	2 hours @ 2375°F (1300°C) Argon			
	24 hours @ 2250°F (1232°C) Vacuum	>1	<0.1	0
	12 hours @ 2250°F (1232°C) Vacuum	2	<0.1	0
	24 hours @ 2250°F (1232°C) Vacuum	2	0.1	0
	4 hours @ 2350°F (1288°C) Vacuum			
	8 hours @ 2250°F (1232°C) Vacuum	1-3	<0.1	0
	6 hours @ 2250°F (1232°C) Vacuum	1-2	<0.1	0
	4 hours @ 2250°F (1232°C) Vacuum	1-3	<0.1	0
	6 hours @ 2375°F (1300°C) Argon (2)	6	0.2	10
	4 hours @ 2250°F (1232°C) Vacuum	3.5	0.2	0
	4 hours @ 2350°F (1288°C) Argon			
Base + VC	24 hours @ 2350°F (1288°C) Vacuum	3	0.5	0
	24 hours @ 2250°F (1232°C) Vacuum	Abnormal Growth	<0.1	0
	8 hours @ 2250°F (1232°C) Vacuum	"	<0.1	0
	4 hours @ 2350°F (1288°C) Vacuum			
	12 hours @ 2250°F (1232°C) Vacuum	Abnormal	<0.1	0
	4 hours @ 2350°F (1288°C) Vacuum	Growth		
	24 hours @ 2250°F (1232°C) Vacuum	Abnormal	0.1	0
	4 hours @ 2350°F (1288°C) Vacuum	Growth		
	6 hours @ 2250°F (1232°C) Vacuum	"	<0.1	0
	4 hours @ 2250°F (1232°C) Vacuum	8	<0.1	0
	24 hours @ 2150°F (1177°C) Vacuum	8	0	0
	6 hours @ 2250°F (1232°C) Vacuum	Abnormal	<0.1	0
	6 hours @ 2350°F (1288°C) Vacuum	Growth		
	6 hours @ 2250°F (1232°C) Vacuum	Abnormal	0.1	0
	6 hours @ 2350°F (1288°C) Vacuum	Growth		
	2 hours @ 2400°F (1315°C) Vacuum			

(1) Rating system described in text.

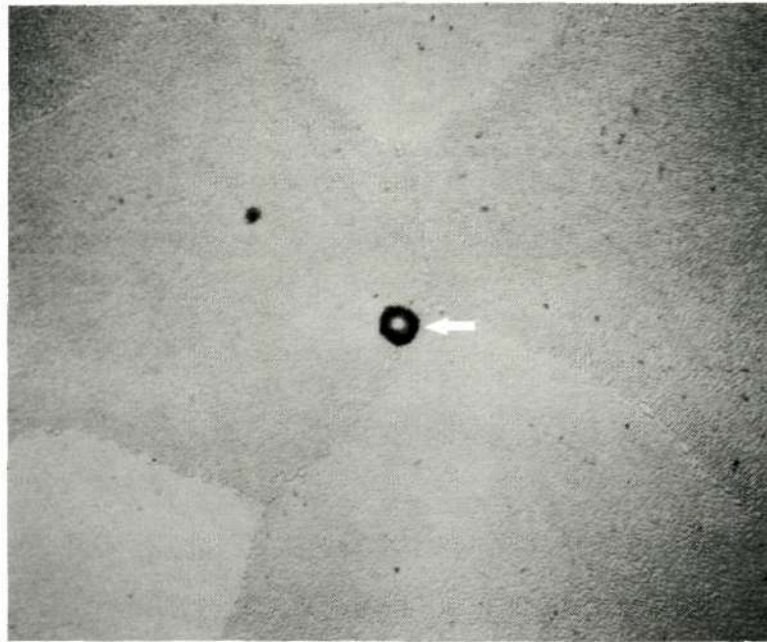
(2) Result from initial 6 hour solution heat treatment, Figure 7.

Table 8

Heat Treatment Cycles Developed for First Series Extruded Alloys
for 1900°F (1038°C)/15,000 psi (104 MN/m²)
Stress Rupture Testing

<u>Alloy</u>	<u>Heat Treatment</u>	<u>ASTM Grain Size</u>	<u>Porosity %</u>
Base	12 hours @ 2250°F (1232°C) vacuum 5 step aging (1)	2	< 0.1
Base + Cr ₃ C ₂	72 hours @ 2250°F (1232°C) vacuum 4 hours @ 2350°F (1288°C) vacuum 5 step aging	6.5	0.1
Base + TiC	8 hours @ 2250°F (1232°C) vacuum 5 step aging	2	< 0.1
Base + VC	6 hours @ 2250°F (1232°C) vacuum 6 hours @ 2350°F (1288°C) vacuum 2 hours @ 2400°F (1315°C) vacuum 5 step aging	90% 2 10% 7	0.1

- (1) Five step aging: 4 hours at 2000°F (1094°C)/6 hours at 1600°F (870°C)/
4 hours at 1800°F (980°C)/24 hours at 1200°F (647°C)/8 hours at 1400°F
(760°C).



Vacuum washed base alloy, 12 hours at 2250°F (1230°C)
in vacuum, 250X, transverse, etched.

Figure 16. Light photomicrograph of vacuum-washed base alloy after 12 hours in vacuum at 2250°F (1230°C) showing that porosity still remains in the microstructure.

This page is reproduced at the
back of the report by a different
reproduction method to provide
better detail.

d. Mechanical Property Results

i. High Temperature Property Results

The 1900°F (1038°C)/15 ksi (104 MN/m²) stress rupture results are listed in Table 9 for all first series alloys, including those for as-extruded, argon heat treated, and vacuum heat treated material for comparison. The best stress rupture life, 22.8 hours, was obtained with base alloy plus TiC. Although all the rupture lives were below that of cast MAR-M246, a gradual improvement was observed for each alloy after vacuum heat treatments. With the exception of the base alloy, however, considerable scatter was observed in the rupture lives. Metallographic evaluation of the failed bars revealed that, although porosity levels were reduced in these specimens, some areas of porosity, many located near the original carbide clusters, were definitely associated with crack initiation. These carbide clusters probably act as nuclei for the coalescence of internal gases. In addition to the porosity, examination near the fracture surface revealed that small grain size was also associated with the fracture path indicating that the response to heat treatment was not homogeneous throughout the extruded bar stock. Examples of these observations are shown in Figures 17 and 18.

Microstructures from the vacuum washed base alloy are shown in Figures 17a and 17b. Note the small grain size, ASTM No. 7, and porosity in Figure 17a and the expected absence of grain boundary precipitation in Figure 17b. Comparison with the grain size results of Table 8 indicated that the heat treatment response in the stress rupture bar was not the same as in the metallographic specimen. Little grain boundary precipitation was expected in the base alloy, Figure 17b, because of the absence of carbon in the alloy. The grain size of the base alloy plus Cr₃C₂, Figure 17c, was similar to that observed in the metallographic specimen, Table 7, but the damaging effect of porosity is evident by the number of cracks emanating from voids. Porosity associated with the original carbide clusters is illustrated for the base alloy plus TiC in Figure 18a. Cracks are also seen to initiate at these voids. The grain size of the base alloy plus VC, Figure 18b, is much smaller than that indicated in Table 7 for the metallographic specimen, and there are several large areas of porosity. In general, the grain boundaries in these specimens had the desired discrete particle type carbide morphology as shown in Figure 18c for the base alloy plus VC.

The tensile results are listed in Table 10 for all fully heat treated first series alloys, including the results for as-extruded material for comparison. As anticipated, in all instances the room temperature and 1400°F (760°C) ultimate and 0.2% offset yield strengths of the fully heat treated material were lower than those of the as-extruded bar stock. This reflected the finer grain size in the as-extruded condition. At 1900°F (1038°C), however, where large grain size is required for high strength values, the fully heat treated material was appreciably stronger, particularly

Table 9

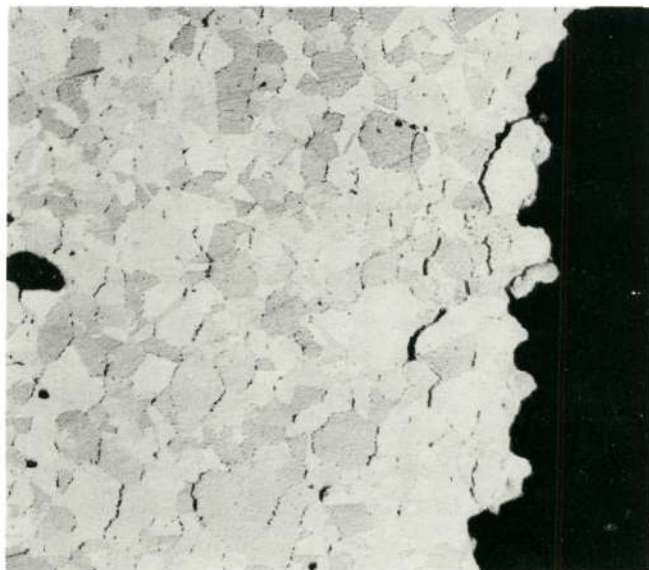
1900°F (1040°C)/15 ksi (104 MN/m²) Stress Rupture Properties for All
Series I Extruded and Heat Treated Alloys

Extrusion No.	Alloy	Heat Treatment	Rupture Life (Hours)	R.A. %	Elongation %
1	Base	As Extruded	0.6	12.7	15.7
			0.4	14.8	17.1
		6 hours @ 2275°F (1246°C) Argon	1.2	1.2	2.0
		6 hours @ 2275°F (1246°C) Argon Full Age (1)	0.9	0.8	2.2
		6 hours @ 2325°F (1274°C) Argon Full Age	1.5	0	1.5
5	Base	Vacuum Washed	3.3	2.8	1.6
		2250°F (1232°C)/12 hours/vacuum Full Age	1.9	4.3	3.1
2	Base + Cr ₃ C ₂	As Extruded	0.1	132.0	67.0
			0.2	65.1	54.5
		72 hours @ 2250°F (1232°C) Vac.	17.44	1.9	1.6
		4 hours @ 2350°F (1288°C) Vac. Full Age	3.70	16.0	15.3
		72 hours @ 2250°F (1232°C) Vac. 4 hours @ 2350°F (1288°C) Vac. Full Age	2.0(2)	1.6	0.8
		100 hours @ 1700°F (925°C)			
3	Base + TiC	As Extruded	1.1	25.8	32.6
			1.0	26.6	31.1
		6 hours @ 2300°F (1260°C) Argon	5.2	1.6	1.7
		6 hours @ 2300°F (1260°C) Argon Full Age	3.9	1.2	2.2
		6 hours @ 2350°F (1288°C) Argon Full Age	3.0	1.6	1.6
		8 hours @ 2250°F (1232°C) Vac. Full Age	7.2 22.82	0.6 1.0	0.8 0
		8 hours @ 2250°F (1232°C) Vac. Full Age	11.5	1.6	3.2
		100 hours @ 1700°F (925°C)			
4	Base + VC	As Extruded	0.5	38.9	40.9
			0.5	40.1	43.9

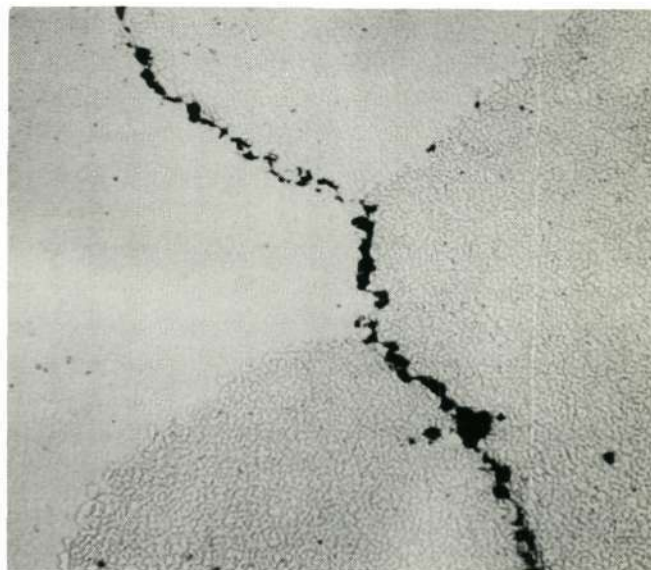
Table 9 (continued)

Extrusion No.	Alloy	Heat Treatment	Rupture Life (Hours)	R.A. %	Elongation %
4	Base + VC	6 hours @ 2250°F (1232°C) Vac.	1.34(2)	0.9	0.4
		6 hours @ 2350°F (1288°C) Vac.	19.6(3)	3.0	7.0
		2 hours @ 2400°F (1315°C) Vac.			
		Full Age			
		2250°F (1232°C)/6 hours/vacuum			
		2350°F (1288°C)/6 hours/ vacuum			
		2400°F (1315°C)/2 hours/ vacuum	16.5	2.0	0.0
		Full Age + 100 hours @ 1700°F (925°C)			
	MAR-M246(4)	100 hours @ 20,000 psi (104 MN/m ²)			
	Alloy with carbon				

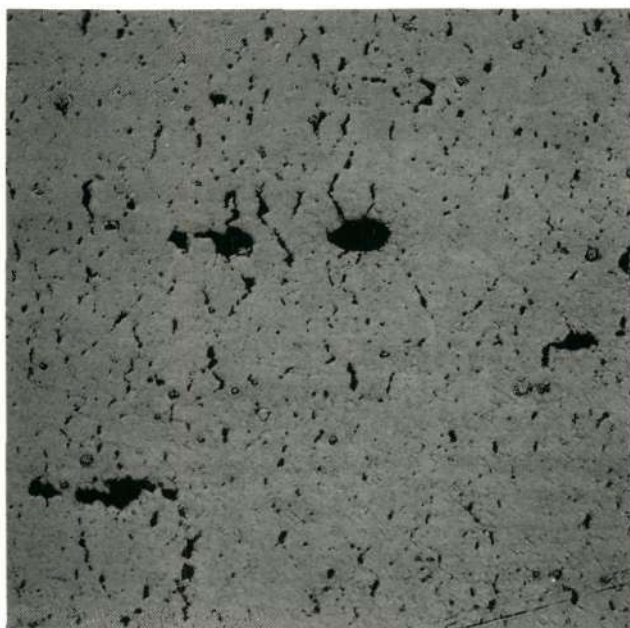
- (1) Full Age: 4 hours @ 2000°F (1094°C) 6 hours @ 1600°F (870°C) 4 hours @ 1800°F (982°C) 24 hours @ 1200°F (647°C) 8 hours @ 1400°F (760°C).
- (2) Specimen broke in the radius, outside the gauge length.
- (3) Furnace temperature dropped to 1870°F (1020°C) during test. Specimen was at least 3.84 hours @ 1900°F (1038°C).
- (4) International Nickel Data Brochure, 1964 Edition.



a) Base alloy, 100X, ASTM grain size No. 7, 1.9 hours life, etched.



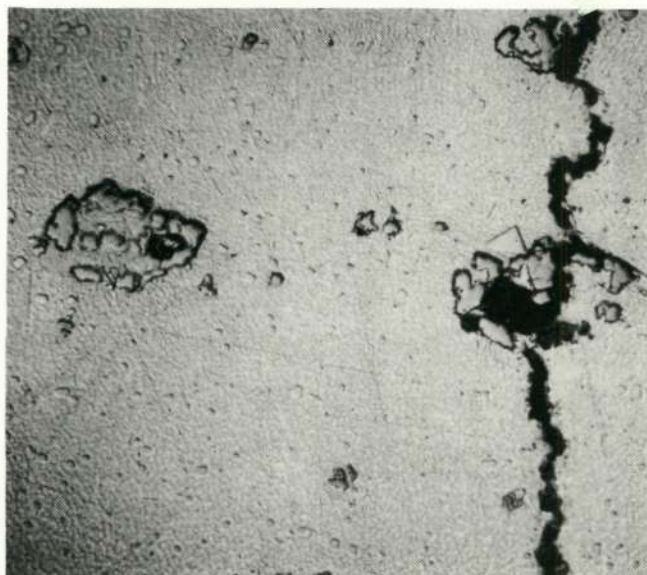
b) Base alloy, 500X, 3.3 hours life, etched.



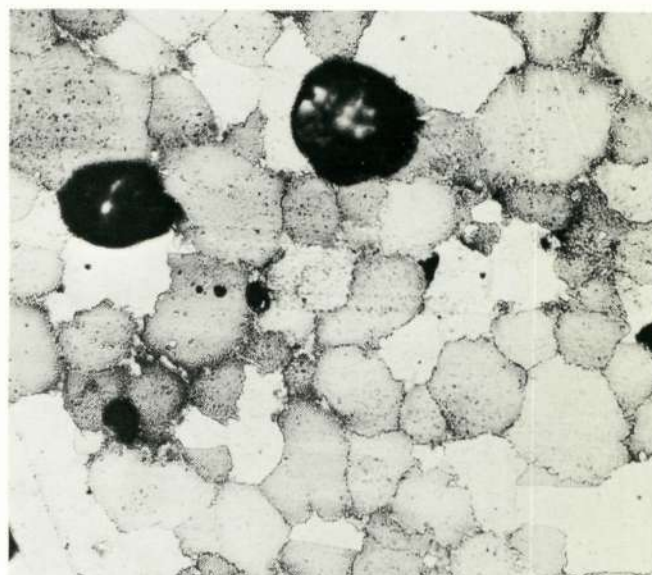
c) Base alloy + Cr_3C_2 , 100X, ASTM grain size No. 7, 3.7 hours life, unetched.

This page is reproduced at the back of the report by a different reproduction method to provide better detail.

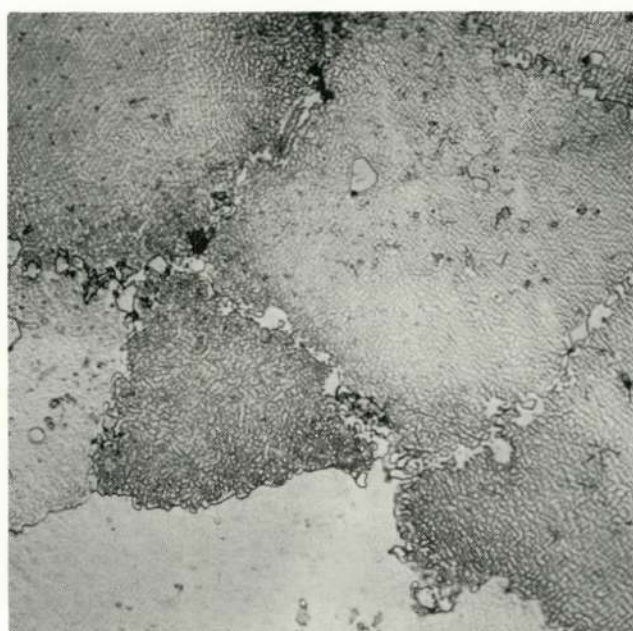
Figure 17. Light photomicrographs of failed 1900°F (1040°C) stress rupture bars of first alloy series, base alloy and base alloy + Cr_3C_2 . Heat treatments and complete stress rupture results are listed in Table 9. All are longitudinal sections.



b) Base alloy + VC, 100X, ASTM grain size No. 3, 16.5 hours life, etched.



b) Base alloy + VC, 100X, ASTM No. 3, 16.5 hours life, etched.



c) Base alloy + VC, 500X, 16.5 hours life, etched.

This page is reproduced at the back of the report by a different reproduction method to provide better detail.

Figure 18. Light photomicrographs of failed 1900°F (1040°C) stress rupture bars of first alloy series, base alloy + TiC and base alloy + VC. Heat treatments and complete stress rupture results are listed in Table 9. All are longitudinal sections.

Table 10

Tensile Properties for Extruded and Heat Treated First Series Alloys

Extrusion No.	Alloy	Heat Treatment	Temperature		Ultimate Strength		0.2% Yield Strength		Elongation %	R.A. %
			(°F)	(°C)	(ksi)	MN/m ²	(ksi)	MN/m ²		
64	1	Base As-Extruded	Room	22	235.9	1627	171.2	1180	14.2	15.7
			1400	760	181.4	1251	168.2	1161	3.1	2.0
			1900	1038	6.1	41.4	4.8	30.6	229.5	90.3
	5	Vacuum Washed 2250°F (1232°C)/13 hours/vac. Full Age (1)	Room	22	208.1	1435	135.9	937	13.2	15.2
			1400	760	151.0	1041	133.1	919	5.1	9.3
			1900	1038	40.6	280	31.8	219	3.6	2.8
	2	Base + Cr ₃ C ₂ As-Extruded	Room	22	218.5	1507	171.0	1179	9.7	11.6
			1400	760	184.4	1241	168.7	1159	5.9	5.1
			1900	1038	6.1	41.4	3.0	20.7	552.9	97.3 (2)
		2275°F (1246°C)/72 hours/vac. 2350°F (1288°C)/4 hours/vac. Full Age	Room(3)	22	102.4	706	NA		1.0	1.2
			1400(3)	760	138.0	952	137.0	945	1.3	1.2
					127.8	881	NA		2.3	1.2
			1900(3)	1038	34.2	236	33.6	232	3.3	1.2
		Base + TiC As-Extruded	Room	22	227.9	1572	171.7	1184	12.7	13.4
			1400	760	195.5	1348	174.6	1204	4.3	5.5
			1900	1038	42.9	296	18.3	126	12.4	10.8
		2250°F (1232°C)/8 hours/vac. Full Age	Room	22	162.3	1119	134.5	928	4.3	6.3
			1400	760	127.8	881	NA		2.3	1.2
					154.0	1062	140.1	967	NA	4.7
			Room	22	55.2	381	42.7	294	2.8	2.4
			1900	1038	55.9	386	43.4	299	3.9	2.4
	4	Base + VC As-Extruded	Room	22	227.1	1567	169.3	1167	11.1	11.7
			1400	760	184.2	1270	170.4	1175	4.6	4.7
			1900	1038	41.6	286	12.4	85.7	18.6	16.4
		2250°F (1232°C)/6 hours/vac. 2350°F (1288°C)/6 hours/vac. 2400°F (1315°C)/2 hours/vac. Full Age	Room	22	112.6	776	NA		1.0	0.4
			1400	760	121.6	839	NA		1.0	1.4
			1900	1038	44.4	306	32.9	227	4.4	3.1
					45.7	315	34.8	240	3.5	2.4

Table 10 (continued)

Extrusion No.	Alloy	Heat Treatment	Temperature		Ultimate Strength		0.2% Yield Strength		Elongation %	R.A. %
			(°F)	(°C)	(ksi)	MN/m ²	(ksi)	MN/m ²		
	Cast		Room	22	140.0	965	125.0	863	5.0	NA
	MAR-M246(4)		1400	760	150.0	1034	125.0	863	5.0	NA
			1800	980	80.0	552	55.0	379	8.0	NA
			1900	1038	62.5	431	37.0	255		
			(5)							

- (1) Full Age: 2000°F (1090°C)/4 hours + 1600°F (870°C)/6 hours + 1800°F (980°C)/4 hours + 1200°F (650°C)/24 hours + 1400°F (760°C)/8 hours.
- (2) Test concluded when specimen extended out of furnace heat zone.
- (3) Subsize specimen: 0.160" (4.1 mm) diameter versus regular 0.252" (6.45 mm) diameter.
- (4) Data from International Nickel Brochure, 1964 Edition.
- (5) Extrapolated data.

the base alloy and the base alloy plus Cr_3C_2 . At 1900°F (1038°C) the base alloy plus TiC exhibited the best ultimate and 0.2% offset yield strengths, while the base alloy plus VC had the next highest values. These were low, however, compared to the estimated values for cast MAR-M246 at the 1900°F (1038°C) - 62.5 ksi (431 MN/m²) ultimate and 37.0 ksi (255 MN/m²) yield strength. At 1900°F (1038°C) the base alloy plus VC exhibited slightly superior ductility. At 1400°F (760°C), however, the base alloy plus TiC offered strength and ductility values comparable to those of cast MAR-M246 while the base alloy plus VC experienced inferior properties, probably because of the high porosity content in the structure, Figure 18b.

ii. Intermediate Temperature Property Results

Material was sent to the NASA Program Manager where it was heat treated specifically for stress rupture testing at intermediate temperatures, 1200°F (650°C) - 1400°F (760°C). The heat treatments and results of these stress rupture tests are listed in Table II compared to cast MAR-M246 and were inferior to cast material. Metallographic examination of the failed stress rupture specimens revealed that porosity in the microstructure was responsible for initiating numerous cracks, Figure 19. The vacuum heat treatments given to these materials were designed to reduce gas porosity in the microstructure and solution the original carbides in order to allow precipitation of desirable hafnium and tantalum rich carbides during subsequent aging treatments. However, the high temperature exposures had to be low enough and short enough to avoid grain growth since this would be deleterious to the stress rupture properties at 1200°F (650°C) and 1400°F (760°C). While the grain size remained small, it was apparent that little of the internal gas porosity was removed by the 4 hour exposure at 2250°F (1230°C) in vacuum, Figure 19.

3. Summary - First Alloy Series

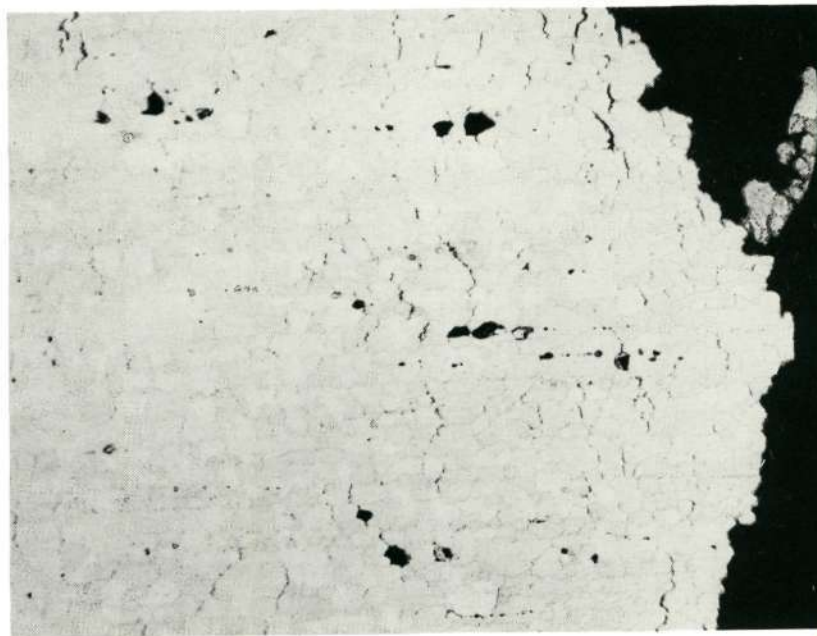
The purpose of evaluating the five extrusions in the first alloy series was to select the metastable carbide Cr_3C_2 , TiC, or VC which was best able to remain intact during consolidation, decompose during high temperature solution treatments, allow grain growth, and finally promote the precipitation of discrete grain boundary carbides during subsequent aging heat treatments. To a certain extent, each of the carbides was able to accomplish this. However, mechanical property tests and metallographic analysis indicated that TiC and VC offered more potential for property optimization than Cr_3C_2 . The best 1900°F (1038°C)/15 ksi (104 MN/m²) stress rupture results were obtained with material containing TiC and VC, even though porosity was evident in the microstructure, Figures 18a and 18b. The base alloy plus TiC had the best 1400°F (760°C) and 1900°F (1038°C) ductility. In addition, base alloy plus VC evidenced the most desirable discrete particle grain boundary carbide morphology, Figure 18c, and both alloys evidenced satisfactory grain size, Table 8.

Table 11

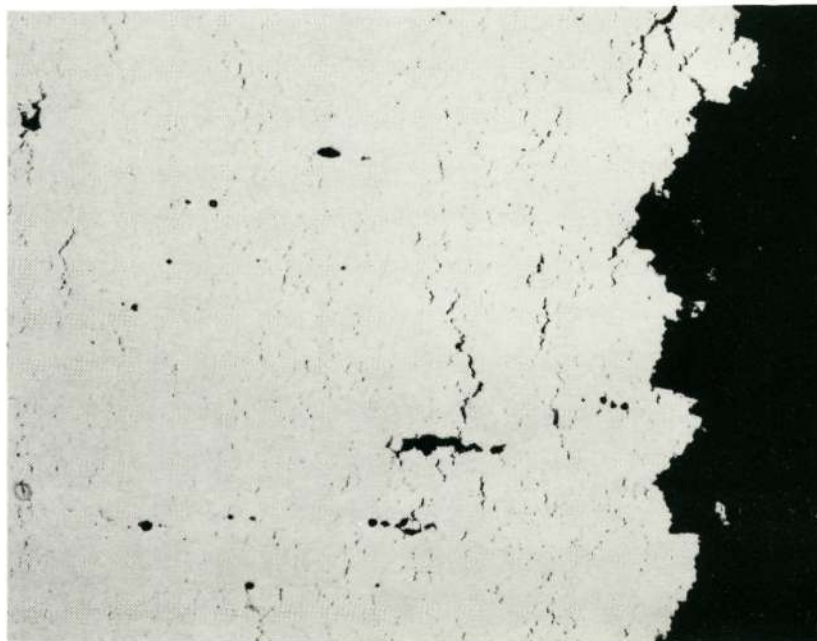
Intermediate Temperature Stress Rupture Data for Extruded and
Heat Treated First Series Alloys

Extrusion No.	Alloy	Heat Treatment	Temperature (°F)	Temperature (°C)	Stress ksi	Level MN/m ²	Life (hours)	Elongation %	R.A. % (1)
2	Base + Cr ₃ C ₂	As Extruded	1200	650	150	1034	104	1.5	0
		4 hours @ 2250°F (1232°C) Vacuum	1200	650	150	1034	16	3	2.5
		4 hours @ 2350°F (1288°C) Vacuum							
		4 hours @ 1975°F (1080°C) Argon							
		48 hours @ 1400°F (760°C) Argon							
		4 hours @ 2250°F (1232°C) Vacuum	1400	760	62	428	1125	8	2.5
		4 hours @ 2350°F (1288°C) Vacuum							
		4 hours @ 1975°F (1080°C) Argon							
		24 hours @ 1600°F (870°C) Argon							
		4 hours @ 2250°F (1232°C) Vacuum	1400	760	62	428	1781	6	5
		4 hours @ 2350°F (1288°C) Vacuum							
		4 hours @ 1975°F (1080°C) Argon							
3	Base + TiC	As Extruded	1200	650	150	1034	5.5	0.8	1.5
		4 hours @ 1975°F (1080°C) Argon	1200	650	150	1034	5	0	2.5
		24 hours @ 1600°F (870°C) Argon							
4	Base + VC	As Extruded	1200	650	150	1034	7.6	1.5	0
		4 hours @ 2250°F (1232°C) Vacuum	1200	650	150	1034	31	3	3
		4 hours @ 2350°F (1288°C) Vacuum							
		4 hours @ 1975°F (1080°C) Argon							
		48 hours @ 1400°F (760°C) Argon							
		4 hours @ 2250°F (1232°C) Vacuum	1400	760	62	428	1243	8	7
		4 hours @ 2350°F (1288°C) Vacuum							
		4 hours @ 1975°F (1080°C) Argon							
		24 hours @ 1600°F (870°C) Argon							
	MAR-M246(1)	Cast	1500	816	62	428	1000		
			1400	760	86	594	1000		

(1) Inco data brochure, 1964 Edition.



a) Base alloy + Cr_3C_2 , 100X, 1781 hours life, etched.



b) Base alloy + VC, 100X, 1243 hours life, unetched.

This page is reproduced at the back of the report by a different reproduction method to provide better detail.

Figure 19. Photomicrographs of failed intermediate temperature stress rupture bars of first series alloys, base alloy + Cr_3C_2 and base alloy + VC. Heat treatments and complete stress rupture results are listed in Table 11. All are longitudinal sections.

Certain specific problem areas were recognized, however. While grain growth was achieved in these alloys, it was not homogeneous from specimen to specimen. Fracture sites were still associated with small grains, Figure 17a, although metallographic analysis of heat treatment specimens established the fact that these same thermal exposures developed much larger grain sizes. In addition, although vacuum heat treatments were successful in reducing the amount of porosity observed throughout the microstructure, it was not removed completely, Figure 18b, and was detrimental to the high temperature properties, Figure 17c. Although it was assumed that argon was the gas entrapped within the original powder particles, Figure 1, neither chemical analysis nor microprobe analysis was able to establish its presence within these voids. Electron microprobe results, shown in Figure 20, do not indicate argon present at the void internal surface.

B. Series 2 - Optimization of Carbon Content

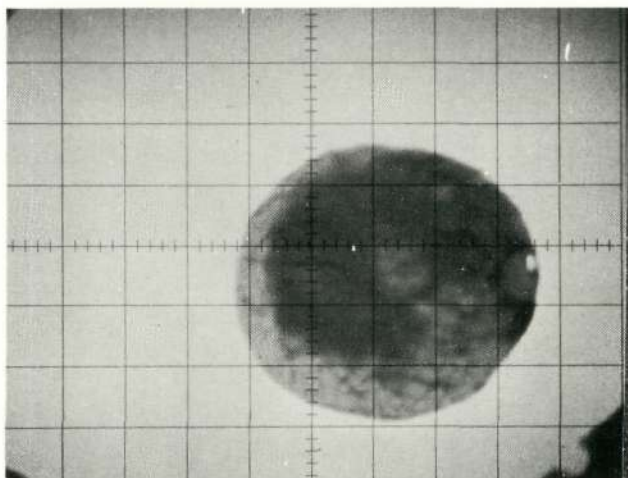
1. Materials and Procedures

a. Powder Production

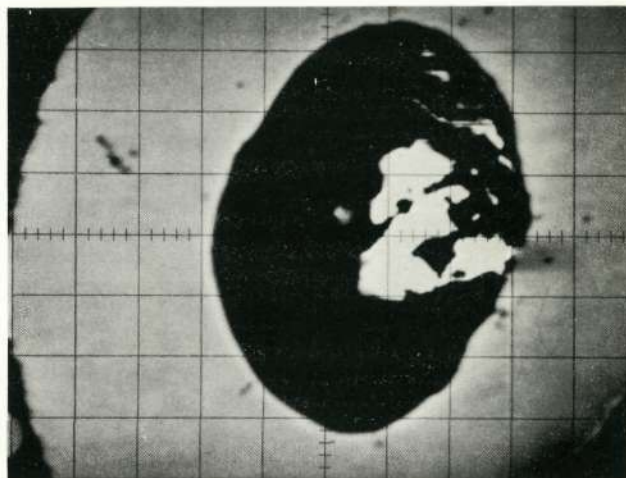
After mechanical property tests and a complete metallographic evaluation of the failed test bars made from the five alloys in the first series of extrusions, VC and TiC metastable carbides were selected for this portion of the program. Since vacuum washing did not eliminate the occurrence of porosity in the heat treated material, it was decided to utilize hydrogen-atomized powder for the remainder of the program. Approximately 36 pounds (16 kg) of -60 +100 mesh and 60 pounds (27 kg) of -100 +325 mesh powder were furnished to TRW by the NASA Program Manager after hydrogen atomization of Cannon-Muskegon's heat VE 226 by Homogeneous Metals, Inc., of Herkimer, New York. The powder was shipped to TRW under a protective nitrogen atmosphere. The hydrogen atomized powder particles were more irregular in shape than the argon-atomized particles and consisted of some flakes as well as droplets, Figure 21. Some surfaces contained protrusions, the dendritic structure was clearly evident, and the secondary dendrite arm spacing somewhat larger than that of the argon-atomized material (6 microns versus 2 microns). There were, however, no indications of porosity within the powder particles themselves.

b. Powder Consolidation

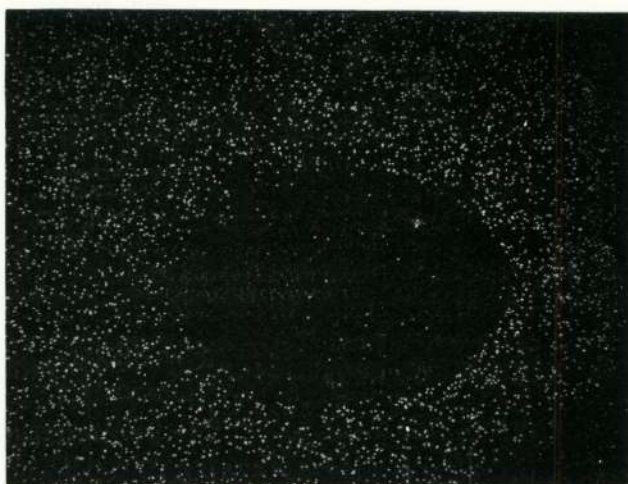
Three alloys were selected for consolidation in this second series. Two alloys contained VC, at the 0.15 and 0.28 w/o carbon levels respectively, to determine the optimum carbon content for this base alloy system. A third alloy, containing TiC at the 0.20 w/o carbon level, was included in order to make a direct comparison with the similar alloy in the first series made from argon atomized powder. In order to increase powder utilization, both -60 +100 and -100 +325 mesh powder sizes were used to make the 8 pound (3.6 kg) extrusions. The constituents for each of the three extrusions are listed below, Table 12.



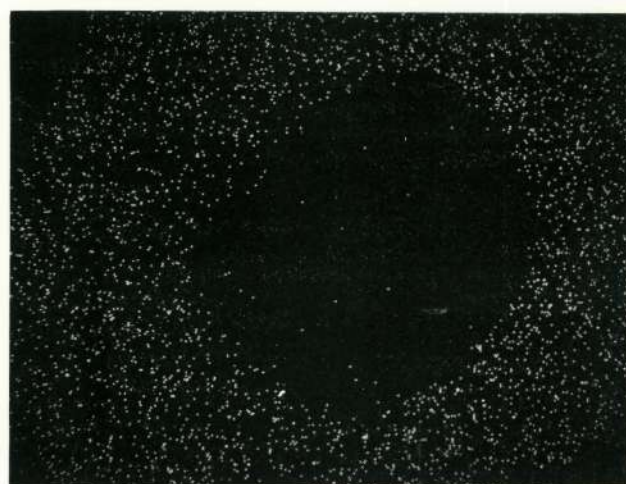
a) Absorbed current image, 800X.



c) Absorbed current image, 800X.



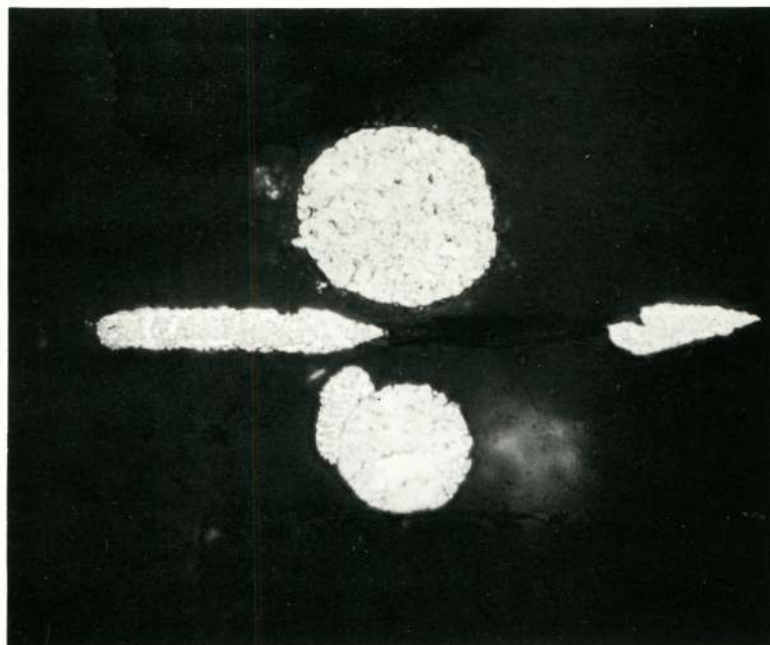
b) Argon raster, 800X.



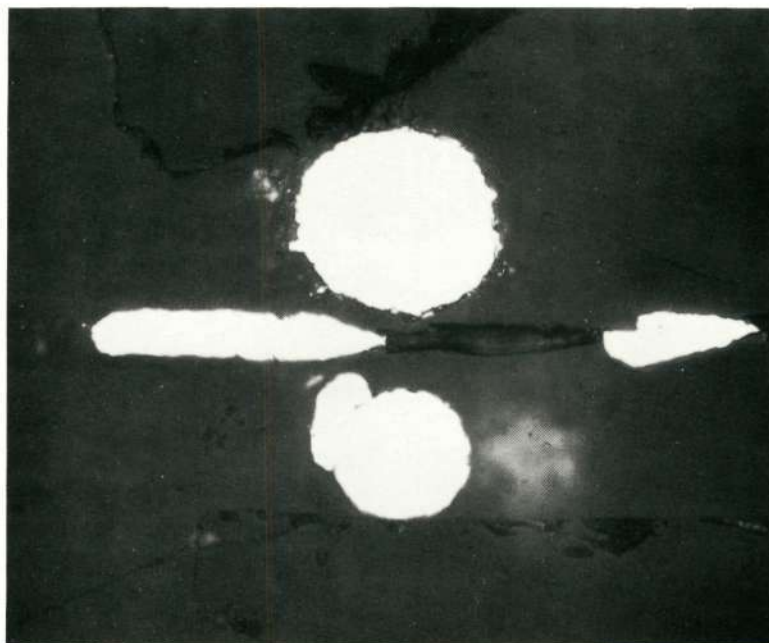
d) Argon raster, 800X.

Figure 20. Electron microprobe photos of as-received argon atomized powder showing results of argon analysis on the internal surfaces of holes observed in powder particles.

This page is reproduced at the back of the report by a different reproduction method to provide better detail.



a) 500X, etched



b) 500X, unetched

This page is reproduced at the back of the report by a different reproduction method to provide better detail.

Figure 21. Light photomicrographs of as-received hydrogen gas atomized MAR-M246 base powder containing no carbon.

Table 12

Constituents of Three Extrusions Made in Second Alloy Series

Extrusion No.	Amount -100 +325 Base Alloy (g)	Amount -60 +100 Base Alloy (g)	Amount Carbide (g)	Percentage Carbon in Carbide	Percentage Carbon in Alloy (Aim)
6	2270	1324	37.2TiC	19.5	0.20
7	2270	1342.4	19.8VC	18.3	0.15
8	2270	1312.4	49.6VC	18.3	0.28

In order to minimize contact with argon, a procedural change was initiated in the blending technique. After loading the various constituents into the TIG welding chamber, it was pumped down to 10^{-3} (1.33×10^{-1} N/m²) and then back filled with nitrogen instead of argon. All the alloy constituents were then sealed in the double conical blender which was removed from the TIG chamber and rotated for 2 hours. After transfer back to the TIG welding chamber, the blended powder was placed into the extrusion cans in an argon atmosphere and sealed. Argon has to be used here since a TIG welding unit operates only in an inert atmosphere. Vacuum washing was again conducted in the electron beam welding chamber at 1000°F (540°C) for 12-16 hours at 10^{-5} (1.33×10^{-3} N/m²) vacuum after which the nose of the extrusion billet was resealed.

The extrusions were made at 2175°F (1190°C) at a 16:1 reduction ratio with the extrusion parameters listed below in Table 13.

Table 13

Extrusion Parameters for Three Extrusions Made in Second Alloy Series

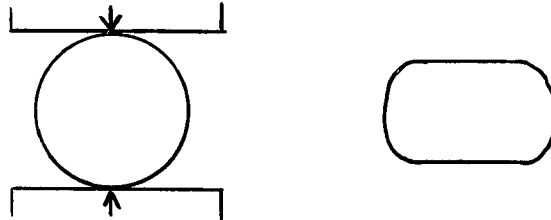
Extrusion No.	Breakthrough Pressure		Ram Speed	
	ksi	MN/m ²	in/sec.	cm/sec.
6	119.5	824	1.7	4.3
7	119.5	824	1.4	3.6
8	110.0	758.4	2.8	7.1

After extrusion, the bar stock was subjected to radiographic, chemical, and light metallographic analysis utilizing procedures as described previously.

c. Property Optimization Procedures

The initial heat treatment studies conducted on these three alloys employed procedures as described previously, including high temperature argon and vacuum treatments and light metallographic evaluation to characterize microstructural features. Since these treatments resulted in a duplex microstructure consisting of both large and small grains, it was decided to evaluate

thermal-mechanical working as a means of promoting a more uniform heat treatment response. Side pressing forging trials were conducted on 1/2 inch (1.27 cm) long, decanned specimens of each alloy. These were first double coated with a propriety formulation for lubrication to offer protection during heat-up to the forging temperature. Side press forging was conducted on a 150 ton Lake Erie hydraulic forging press at temperatures below the recrystallization point for each alloy and deformations of approximately 5% reduction in diameter were imparted in one blow between flat dies.



After determining at what temperature these reductions were possible, 3 inch (7.6 cm) long specimens of each alloy were side pressed and sent to NASA for heat treatment in their gradient annealing furnace, the procedure for the operation of which has been described previously (2). The specimens were annealed for 24 hours in a thermal gradient ranging from 2400°F (1315°C) to 2000°F (1090°C) and returned to TRW for metallographic evaluation.

After selection of the heat treatment promoting the desired grain growth, specimens of each alloy were side pressed, fully heat treated, machined and tested for room temperature, 1400°F (760°C), and 1900°F (1038°C) tensile properties and 1900°F (1038°C)/15 ksi (104 MN/m²) stress rupture properties utilizing procedures described previously. After testing, all specimens were evaluated metallographically to determine locations of fracture initiation.

2. Results and Discussion

a. Extrusion Bar Inspection

i. Radiographic and Chemical Analysis

All three alloys were successfully extruded at 2175°F (1190°C) and radiographic inspection indicated no defects in the extruded bar stock. The chemical analyses for the three extrusions are presented in Table 14 along with the original vacuum-melted billet chemistry (Cannon-Muskegon Heat No. VE-226) and the chemistry of the as-received base alloy powder. The target 0.20 w/o carbon content for the base alloy plus TiC was successfully achieved. The carbon levels for the material containing VC were slightly higher than desired. Since VC additions were employed to optimize the carbon content in the alloy, these deviations were acceptable because they were both above and below the 0.2 w/o level of the base plus VC alloy studied in the first alloy series. Similar to the results for the first series of alloys, the molybdenum contents were lower than the target range, Table 2, but this reflected the lowered molybdenum content of the as-received powder. For both alloys containing VC, the zirconium contents were also above the target range. The other important deviation from the target range was the oxygen content, which was 120 ppm in the as-received powder and increased as a result of the hot extrusion consolidation process.

Table 14

Chemical Analyses of Billet, Atomized Powder and Extruded Material
of Second Alloy Series

<u>Material</u>	<u>C</u>	<u>Cr</u>	<u>Mo</u>	<u>W</u>	<u>Co</u>	<u>Ti</u>	<u>Al</u>	<u>B</u>	<u>Zr</u>	<u>Ta</u>	<u>Hf</u>	<u>V</u>	<u>O</u>	<u>N</u>
Ingot (Cannon- Muskegon Heat VE-226)	.01	8.86	2.50	9.98	9.87	1.45	5.53	0.016	0.05	1.80	.87	-	.0095	-
As Atomized Base Powder	.006	9.22	1.62	11.80	9.36	1.24	4.85	0.015	0.053	1.73	.67	-	.0120	-
Base + TiC 0.2%C	0.20	9.29	1.47	10.14	9.66	1.72	5.09	0.012	0.08	1.97	.43	-	.0210	.0080
Base + VC 0.1%C	0.15	9.02	1.46	10.5	9.12	1.63	5.17	0.014	0.10	1.92	.43	.87	.0167	.0060
Base + VC 0.25%C	0.28	8.96	1.47	10.6	8.97	1.57	5.23	0.015	0.15	1.71	.57	1.80	.0130	.0050
MAR-M246 Nominal Analysis	0.15	9.0	2.50	10.0	10.0	1.50	5.5	0.015	0.05	1.5	-	-	-	-

ii. Metallographic Analysis

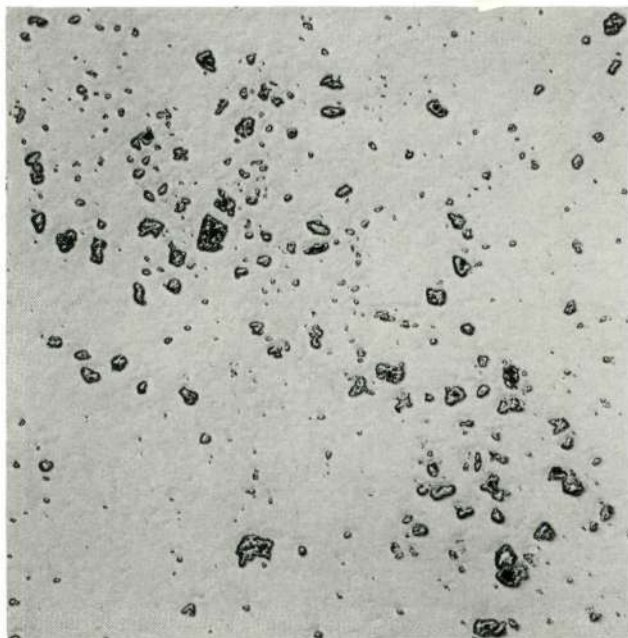
Metallographic analysis of the as-extruded microstructure revealed that the distribution of the original carbides varied throughout the extrusion and that there were some areas of incomplete compaction, Figure 22. Figures 22a and 22b were taken from the nose portion of the base alloy plus VC (0.28 w/o C) and show considerable variation in carbide distribution. Inhomogeneity in heat treatment response could be expected from this type of microstructure because solution heat treatments do not completely solution or decompose the original carbides which could act to pin grain boundary movement in certain areas. Incomplete compaction, Figure 22c, would be expected to have the same detrimental effect on high temperature properties as gas porosity.

b. Solution and Grain Growth Heat Treatment Study

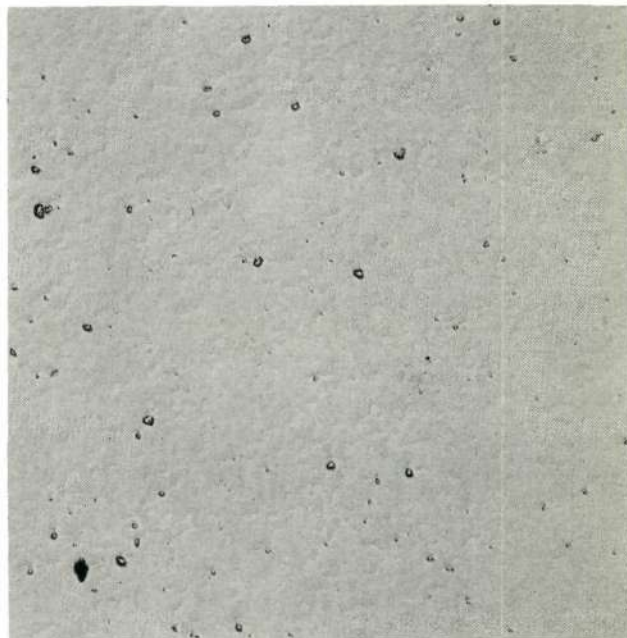
The preliminary heat treatment results for these hydrogen-atomized powders indicated no gas porosity in the microstructure but a deviation in response from that experienced with argon-atomized powder for identical heat treatments. The same vacuum heat treatment, as was applied to the argon-atomized powder, 8 hours at 2250°F (1232°C) to achieve an ASTM No. 2 grain size, was employed in the initial heat treatment of the hydrogen atomized TiC containing material. No gas porosity was observed but abnormal grain growth occurred. Certain grains grew at the expense of smaller grains throughout the matrix. The result was a duplex grain size which is considered detrimental for high temperature stress rupture strength since the smaller grains in the matrix are areas of weakness.

For the alloy containing VC a grain growth cycle consisting of 6 hours/2250°F (1232°C) + 6 hours/2350°F (1288°C) + 2 hours/2400°F (1360°C) in vacuum produced a duplex structure of approximately 90% ASTM No. 2 grains, the remainder ASTM No. 3-6, in the argon-atomized alloy containing 0.2 w/o carbon. The hydrogen-atomized alloy containing VC at 0.15 w/o carbon, exposed to 6 hours/2250°F (1232°C) + 6 hours/2350°F (1288°C) in argon exhibited incipient melting. The hydrogen-atomized alloy containing VC at 0.28 w/o carbon was exposed 24 hours at 2350°F (1288°C) in vacuum resulting in an ASTM No. 4.5 grain size with uniform grains and no incipient melting. No gas porosity was observed in the hydrogen-atomized material after heat treatment.

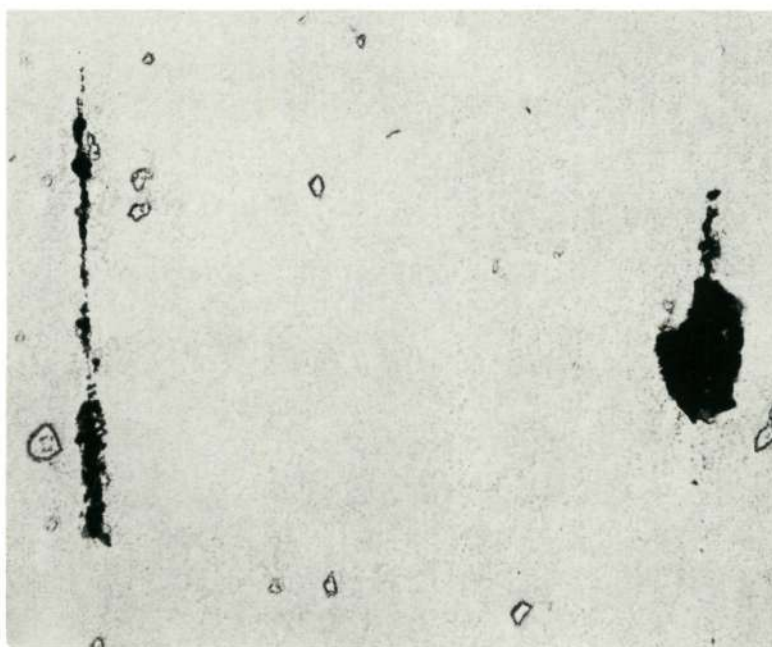
A heat treatment study was then conducted to promote an ASTM No. 2-3 grain size in these alloys by applying homogenization treatments below the incipient melting temperature followed by treatments above this temperature. Since gas porosity was not observed in the heat treated microstructure of the hydrogen-atomized material, all subsequent heat treatments were conducted in an argon atmosphere. The results of this heat treatment study are listed in Table 15.



a) Base + VC (0.28 w/o C), nose section, 100X, unetched.



b) Base + VC (0.28 w/o C), nose section, 100X, unetched.



c) Base + TiC (0.20 w/o C) mid section, 100X, etched.

This page is reproduced at the back of the report by a different reproduction method to provide better detail.

Figure 22. Light photomicrographs of as-extruded alloys at 100X, etched, in the longitudinal section. Note variation in the distribution of original carbides (a) and (b), and incomplete compaction (c).

Table 15

Metallographic Results of Solution Heat Treatments Applied
to Alloys of Series 2

<u>Extrusion No.</u>	<u>Alloy</u>	<u>Heat Treatment</u>	<u>Grain Size</u>
6	Base + TiC (0.2 w/o C)	8 hours @ 2250°F (1232°C) Vacuum	ASTM No. 2
		8 hours @ 2250°F (1232°C) Argon	Secondary Recrystallization
		4 hours @ 2250°F (1232°C) Argon	Secondary Recrystallization
		12 hours @ 2250°F (1232°C) Argon	Secondary Recrystallization
		4 hours @ 2275°F (1246°C) Argon	ASTM No. 7
		24 hours @ 2275°F (1246°C) Argon	ASTM No. 7
		24 hours @ 2300°F (1260°C) Argon	ASTM No. 6
		24 hours @ 2325°F (1274°C) Argon	ASTM No. 6
		24 hours @ 2350°F (1288°C) Argon	Incipient Melting
		24 hours @ 2325°F (1274°C) Argon	ASTM No. 4
		24 hours @ 2340°F (1282°C) Argon	
		24 hours @ 2325°F (1274°C) Argon	ASTM No. 4
		24 hours @ 2340°F (1282°C) Argon	
		24 hours @ 2350°F (1288°C) Argon	
		24 hours @ 2325°F (1274°C) Argon	ASTM No. 4
		24 hours @ 2340°F (1282°C) Argon	
		24 hours @ 2350°F (1288°C) Argon	
		24 hours @ 2360°F (1293°C) Argon	
7	Base + VC (0.15 w/o C)	6 hours @ 2250°F (1232°C) Vacuum	90% ASTM No. 2 + 10% ASTM No. 3-6
		6 hours @ 2350°F (1288°C) Argon	
		2 hours @ 2400°F (1315°C) Argon	
		6 hours @ 2250°F (1232°C) Argon	Secondary Recrystallization
		2 hours @ 2250°F (1232°C) Argon	Secondary Recrystallization
		4 hours @ 2250°F (1232°C) Argon	Secondary Recrystallization
		6 hours @ 2250°F (1232°C) Argon	Incipient Melting
		6 hours @ 2350°F (1288°C) Argon	

Table 15 (continued)

Extrusion No.	Alloy	Heat Treatment	Grain Size
7	Base + VC (0.15 w/o C)	6 hours @ 2300°F (1260°C) Argon	ASTM No. 7
		24 hours @ 2325°F (1274°C) Argon	Secondary Recrystallization
		24 hours @ 2350°F (1288°C) Argon	Incipient Melting
		24 hours @ 2300°F (1260°C) Argon	Incipient Melting
		24 hours @ 2350°F (1288°C) Argon	
		24 hours @ 2300°F (1260°C) Argon	ASTM No. 1-4
		24 hours @ 2340°F (1282°C) Argon	
		24 hours @ 2300°F (1260°C) Argon (2)	ASTM No. 1.5
		24 hours @ 2340°F (1282°C) Argon	
		24 hours @ 2350°F (1288°C) Argon	
8	Base + VC (0.28 w/o C)	6 hours @ 2250°F (1232°C) Argon	ASTM No. <8
		8 hours @ 2325°F (1274°C) Argon	ASTM No. 8
		8 hours @ 2300°F (1260°C) Argon	ASTM No. 8
		24 hours @ 2350°F (1288°C) Argon	ASTM No. 5
		24 hours @ 2375°F (1300°C) Argon	Incipient Melting
		24 hours @ 2350°F (1288°C) Argon	ASTM No. 4
		24 hours @ 2375°F (1300°C) Argon	
		24 hours @ 2350°F (1288°C) Argon	ASTM No. 4
		24 hours @ 2375°F (1300°C) Argon	
		24 hours @ 2385°F (1310°C) Argon	
		24 hours @ 2350°F (1288°C) Argon	ASTM No. 3.5
		24 hours @ 2375°F (1300°C) Argon	
		24 hours @ 2385°F (1310°C) Argon	
		24 hours @ 2395°F (1313°C) Argon	

(1) Heat treatment developed from first set of extrusions, made with argon atomized powder.

(2) Selected as most optimum heat treatment.

For the base alloy plus VC (0.15 w/o C) a three step solution heat treatment consisting of 24 hours at 2300°F (1260°C) + 24 hours at 2340°F (1280°C) + 24 hours at 2350°F (1288°C) produced an ASTM No. 1.5 grain size and was selected as the optimum grain growth treatment for this alloy. For the base alloy plus TiC (0.2 w/o C) and the base alloy plus VC (0.28 w/o C) heat treatments consisting of four different exposures produced grain sizes of ASTM No. 4 and 3.5 respectively. These grain sizes were below the desired ASTM No. 2 considered adequate for good stress rupture properties.

c. Thermal Mechanical Process Study

Results of the solution heat treat study conducted on the three extruded alloys of Series 2 indicated that the desired ASTM No. 2 grain size was developed only in the base alloy plus VC (0.15 w/o C). For the other two alloys, however, multiple solution treatments produced smaller grain sizes considered inadequate for good stress rupture properties. Additional exposures at higher temperatures may have resulted in the desired grain size, but a complex high temperature heat treatment involving four or more steps would have been required making the process undesirable from an industrial standpoint. In addition, metallographic analysis revealed that in spite of the numerous high temperature treatments, all of the original carbides were not completely solutioned. With the degree of variation in carbide distribution throughout the microstructure exhibited by the extruded material, Figure 22, a completely homogeneous grain growth response could not be assured.

In order to simplify the complex solution heat treatments and promote a more uniform heat treatment response, thermal-mechanical processing consisting of side press forging to impart a uniform controlled amount of deformation into the structure followed by high temperature heat treatments for grain growth was investigated. Since all three alloys exhibited a recrystallized structure after hot extrusion at 2175°F (1190°C) a forging temperature of 2150°F (1175°C) was selected. Specimens of each alloy were reduced 5% in diameter in one blow by side pressing (flat forging) in a 150 ton Lake Erie hydraulic press.

Side pressed material from the base alloy plus TiC (0.2 w/o C) and the base alloy plus VC (0.28 w/o C) was sent to the NASA Program Manager and heat treated for 24 hours in a thermal gradient furnace with a temperature gradient of 2000°F (1095°C) - 2400°F (1315°C). Metallographic examination revealed that an ASTM grain size of No. 2 was achieved in the base alloy plus TiC (0.2 w/o C) after 24 hours at 2350°F (1288°C), while ASTM No. 3 was achieved in the base alloy plus VC (0.28 w/o C) after 24 hours at 2400°F (1315°C).

Utilizing these results, a heat treatment was selected for each of the three alloys which produced the desired ASTM No. 2-3 grain size in the microstructure and is listed below:

Base alloy + TiC (0.2 w/o C): side press + 24 hours at 2355°F (1290°C)A

Base alloy + VC (0.15 w/o C): 24 hours at 2300°F (1260°C)A
+ 24 hours at 2340°F (1280°C) + 24 hours at 2350°F (1288°C)A

Base alloy + VC (0.28 w/o C): side press + 24 hours at
2400°F (1315°C) vacuum

The microstructures of material receiving these thermal mechanical treatments are shown in Figures 23a, 23b, and 23c.

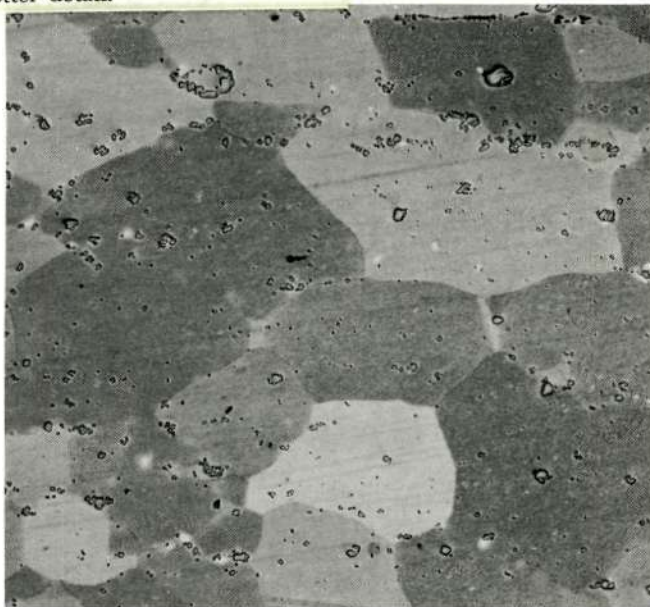
d. Mechanical Property Results

The mechanical property results for the second series alloys including 1900°F (1038°C) 15 ksi (104 MN/m²) stress rupture and room temperature, 1400°F (760°C), and 1900°F (1038°C) tensile tests are listed in Tables 16 and 17, respectively. For the stress rupture tests, several different treatments were evaluated for each alloy, each being developed from the thermal-mechanical processing results or the high temperature solution treatment studies. Side pressing was also used for the alloy containing VC at the 0.15 w/o C level because nonuniform grain size was observed in specimens heat treated without the prior forging operation. The tensile properties listed in Table 17 were obtained for treatments exhibiting the best stress rupture properties. The complex five step aging heat treatment utilized for the first series alloys was replaced by a two step sequence consisting of 4 hours at 1975°F (1080°C)A plus 24 hours at 1700°F (925°C)AC which produced a fine intragranular gamma-prime precipitate and a discrete particle carbide morphology in the grain boundaries as shown in Figure 23d.

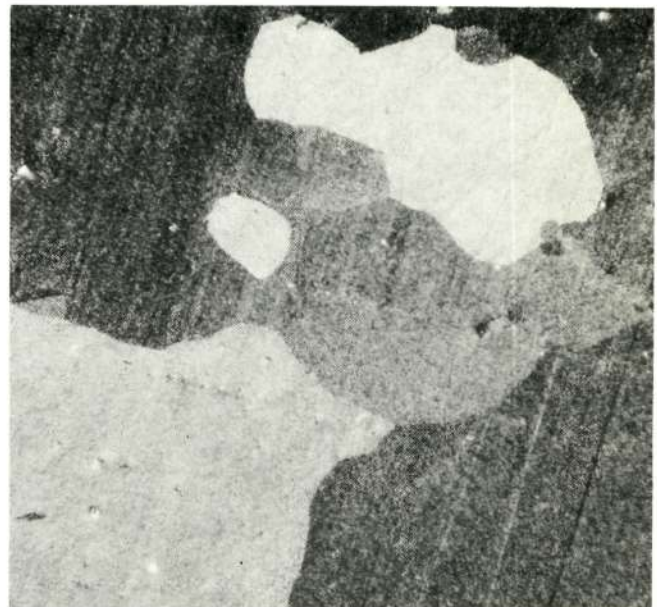
The base alloy plus VC (0.28 w/o C) exhibited the best stress rupture lives while the base alloy plus TiC (0.20 w/o C) exhibited the best rupture ductility. The data indicated that increased amounts of carbon enhanced rupture life, but also slightly lowered rupture ductility. All of the stress rupture lives were low in comparison to cast MAR-M246. The tensile properties of Table 17 substantiated the fact that higher carbon content promoted increased tensile strength, but did not indicate any deleterious effect on tensile ductility. The base alloy plus VC (0.28 w/o C) exhibited superior tensile properties. All of the tensile properties were, however, inferior to those of case MAR-M246.

Comparison of these stress rupture data to those of Table 9 for the first series alloys indicated more consistency in the second series alloys. For similar alloys at the same carbon content, base alloy plus TiC (0.2 w/o C), the maximum rupture life was slightly higher, 26.2 hours in the second series versus 22.8 hours for the first series. The hydrogen-atomized material had a greater rupture ductility. For the material containing VC, the lower carbon content hydrogen-atomized material (0.15 w/o C) had a maximum rupture life of 22.0 hours versus 16.5 hours for argon-atomized material (0.20 w/o C), and the ductility was slightly better. The hydrogen-atomized material at higher carbon content (0.28 w/o C) had substantially greater stress rupture life, 51.7 hours. Comparison of the second series' tensile properties, Table 17, with those of the first series, Table 10, indicated that, in general, the second series were inferior.

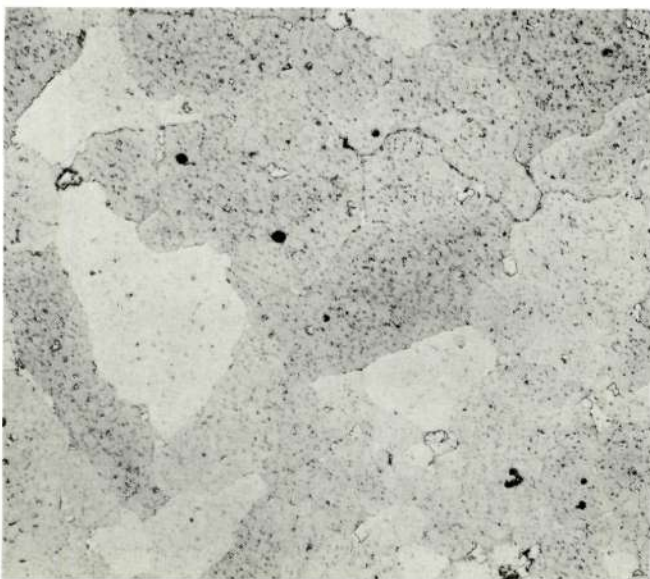
This page is reproduced at the back of the report by a different reproduction method to provide better detail.



a) Base alloy + TiC (0.2 w/o C), side press + 24 hours at 2355°F (1290°C), ASTM grain size No. 2-3, 100X, unetched.



b) Base alloy + VC (0.15 w/o C), 24 hours at 2300°F (1260°C) A + 24 hours at 2340°F (1282°C) A + 24 hours at 2350°F (1288°C) A, ASTM grain size No. 1-3, polarized light, 100X, unetched.



c) Base alloy + VC (0.28 w/o C), side press + 24 hours at 2400°F (1315°C), vacuum, ASTM grain size No. 2-3.



d) Base alloy + VC (0.28 w/o C) side press + 24 hours at 2400°F (1315°C) vacuum + 1975°F (1090°C) A + 24 hours at 1700°F (927°C) air cool, 500X, etched.

Figure 23. Photomicrographs of extruded and side pressed Series 2 alloys after grain growth heat treatment, (a), (b) and (c) and after low temperature aging, (d).

Table 16

1900°F (1040°C)/15 ksi (104 MN/m²) Stress Rupture Results
for Series 2 Alloys

Extrusion No.	Alloy	Heat Treatment	Life (Hours)	Elongation %	R.A. %	ASTM Grain Size No.	Larson-Miller Parameter (4)
6	Base + TiC (0.2 w/o C)	Side Press (1) +	26.2	3.8	3.1	4-5	50.5
		2355°F (1290°C)/24/A	22.4	4.2	2.7	5-6	50.3
		+ Age (2)					
		Side Press + 2355°F (1290°C)/24/A +	4.9	0.6	0.4	3-5	48.7
		2375°F (1300°C)/24/A + Age	25.5	1.4	2.4	3-5	50.4
7	Base + VC (0.15 w/o C)	2300°F (1260°C)/24/A	2.9	0.6	-	3-4	48.0
		+ 2340°F (1282°C)/24/A	2.9	0.9	0.6	3-4	48.0
		+ 2350°F (1288°C)/24/A	1.8	2.3	1.9	6	47.6
		+ Age	1.2	-	-	5-6	47.2
		Side Press + 2345°F (1285°C)/24/Vac.	12.2	1.8	1.2	3-4	49.8
		+ 2355°F (1290°C)/24/Vac. + Age	21.7	2.3	1.6	3-5	50.2
		Side Press + 2355°F (1290°C)/24/Vac.	22.1	2.4	1.6	3-4	50.3
		+ 2365°F (1295°C)/24/Vac. + Age	20.7	2.8	1.6	3-5	50.1
8	Base + VC (0.28 w/o C)	Side Press + 2400°F (1315°C)/24/Vac.	51.7	0.6	1.2	2-4	51.1
		+ Age	47.9	2.1	2.0	2-4	51.0
		Side Press + 2400°F (1315°C)/48/Vac.	26.3	2.0	0.8	1-2	50.5
		+ Age	16.2	1.6	0.4	1-2	50.0
	Cast MAR-M246(3)		100			2-3	51.9

(1) Side Press - 5% reduction at 2150°F (1175°C).

(2) Age: 1975°F (1080°C)/4/A + 1700°F (925°C)/24/AC.

(3) Inco data brochure, 1964 Edition

(4) Larson Miller Parameter $P = T(20 + \log t)$

Table 17

Tensile Properties of Second Series Alloys

Extrusion No.	Alloy	Condition	Temperature		Ultimate Strength		0.2% Yield Strength		Elongation %	R.A. %
			(°F)	(°C)	ksi	MN/m ²	ksi	MN/m ²		
8	Base + VC (0.28 w/o C)	Side Press(1) +	Room	22	126.0	870	-		0.8	1.2
		2355°F (1290°C)/	1400	760	116.8	805	-		1.6	1.0
		24/A + Age(2)	1900	1038	48.7	335	42.3	292	1.2	1.2
7	Base + VC (0.15 w/o C)	Side Press +								
		2355°F (1290°C)/	1400	760	80.8	57	-		1.6	1.8
		24/Vac. + 2365°F (1295°C)/24/Vac. + Age	1900	1038	43.3	298	41.5	286	1.0	
6	Base + TiC (0.20 w/o C)	Side Press +	1400	760	98.0	675	-		1.3	0.8
		2355°F (1290°C)/	1900	1038	44.5	307	38.7	267	1.8	0.8
		24/A + 2375°F (1300°C)/24/A + Age								
	MAR-M246(3)	Cast	Room	22	140	965	125	862	5.0	NA
			1400	760	150	1034	125	862	5.0	NA
			1800	980	80	552	55.0	379	8.0	NA
			1900(4)	1038	62.5	431	37.0	255		

- (1) Side press = 2150°F (1175°C) 5% reduction.
 (2) Age = 1875°F (1080°C)/4/A + 1700°F (925°C)/24/AC.
 (3) International Nickel Data Brochure, 1964 Edition.
 (4) Extrapolated values.

A plot of the maximum rupture lives for each alloy in both series is shown in Figure 24. Included in this figure are results for as-extruded material, argon and vacuum heat treated material, and thermally-mechanically processed material. Although the results for the best alloy, thermal-mechanically processed hydrogen-atomized base alloy plus VC (0.28 w/o C), are below those for cast MAR-M246, a steady improvement in rupture life is apparent. Stress rupture and tensile data for other powder alloys tested at comparable temperatures are listed in Tables 18 and 19, respectively (2-12). Stress rupture lives for these alloys and the base alloy plus VC (0.28 w/o C) are shown in the Larson-Miller plot of Figure 25. It was concluded that the alloy containing VC exhibited the best combination of high temperature stress rupture and tensile properties reported for conventional powder metallurgy alloys to date with the possible exception of results reported by NASA for Inco 713C (2). It should be noted that data collected in the present program were obtained on tests conducted at 1900°F (1038°C), whereas the highest temperature the Inco 713C was tested at was 1800°F (980°C). For a direct comparison to be made between the two materials, the Inco 713C should be tested at 1900°F (1038°C).

e. Metallographic Analysis of Failed Test Bars

Metallographic analysis of the failed test bars revealed three important factors which may contribute to lower than optimum property levels in the second alloy series: variable grain size, incomplete compaction, and an embrittling angular carbide phase. Grain size measurements listed in Table 16 with the stress rupture results indicated that the best rupture lives were obtained with base plus VC (0.28 w/o C) material exhibiting an ASTM 2-4 grain size. In many instances specimens exhibiting lower rupture lives had a much finer grain structure. Representative microstructures for large grained and fine grained material are shown in Figure 26. The fine grain size exhibited by many of the rupture bars illustrated the fact that although the rupture properties of the second alloy series were more reproducible than those of the first alloy series, grain growth response was still inhomogeneous in spite of the application of thermal mechanical working.

Large grain size was a necessary but not sufficient requirement for good stress rupture properties. As shown in Figure 27, material which exhibited fairly good grain size had poor rupture properties possibly because of incomplete compaction. It is believed that incomplete compaction resulted from utilization of larger mesh size hydrogen atomized powder. The single extrusion at 16:1 reduction ratio was insufficient to promote complete compaction.

The third microstructural feature which could have contributed to the low property levels exhibited by the Series 2 alloys was an embrittling angular carbide. This constituent was observed throughout the microstructure after the decomposition of the original carbides. Because of efforts to reduce gas porosity and obtain an ASTM No. 2 grain size, the importance of this phase was overlooked throughout the Series 1 evaluation. The embrittling nature of this carbide is illustrated in Figure 28 for alloys

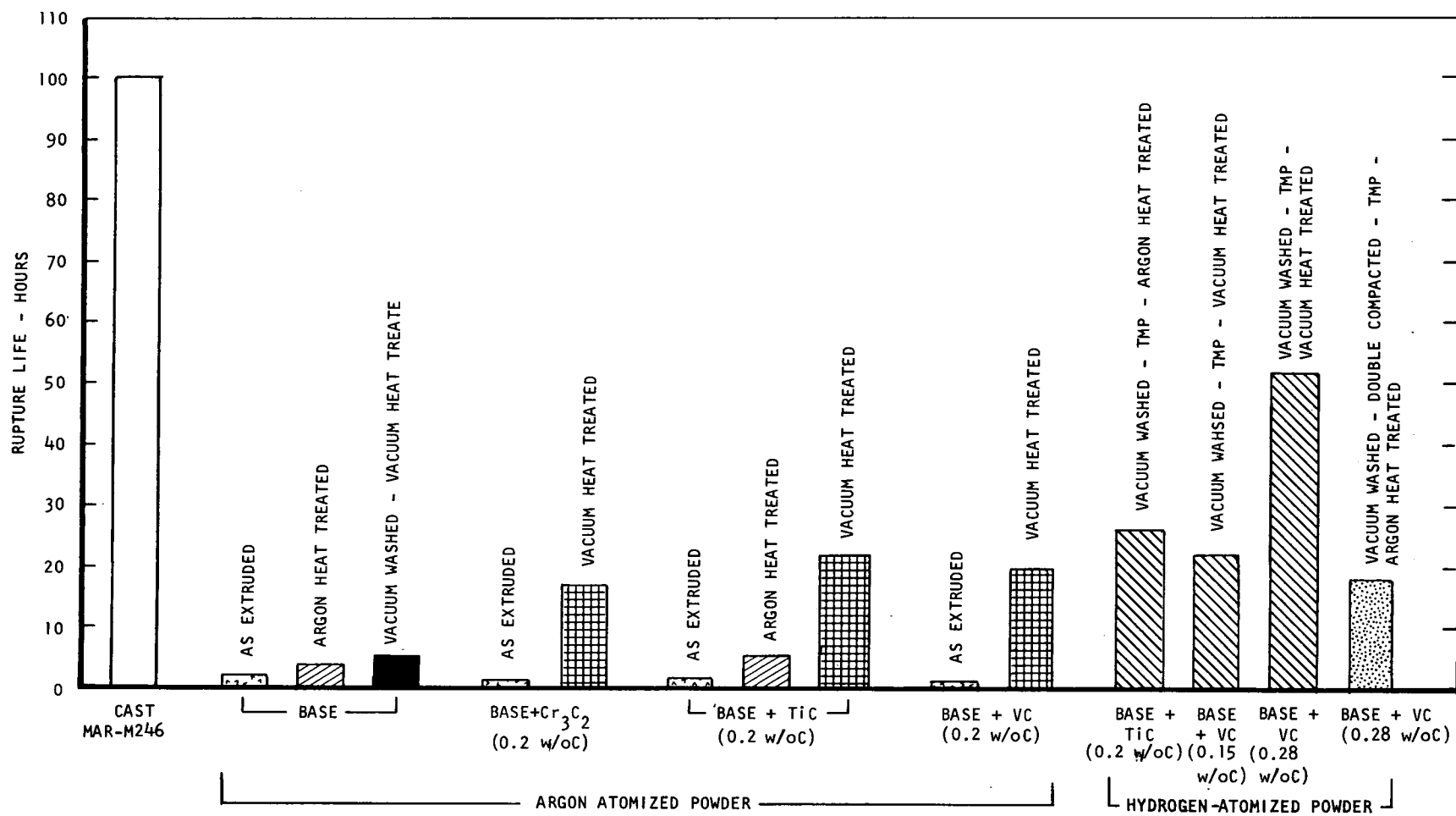


Figure 24. Maximum 1900°F (1040°C)/15 ksi (104 MN/m²) stress rupture lives for carbide blended superalloy powder alloys compared to cast MAR-M246.

Table 18

Stress Rupture Properties of Superalloy Powder Metallurgy Alloys

Source	Alloy	Condition	Temperature		Stress		Life (Hours)	Elongation %	Larson-Miller Parameter (1)
			(°F)	(°C)	ksi	MN/m ²			
NASA (3)	VI-A	Extrude + Heat Treat	1800	980	7.5	51.7	4.0	300	45.2
NASA (3)	VI-A	Extrude + Autoclave Heat Treat	1800	980	7.5	51.7	756.1	330	51.4
NASA (6)	713C	Extrude + Heat Treat	1900	103	10	68.9	98.1	4	51.8
NASA (6)	TAZ8A	As Extruded	1900	103	10	68.9	4.1	600	48.6
			2000	1090	2	13.8	0.1	-	46.7
		Extrude + Heat Treat	1900	103	15	104	2.2	-	48.0
Battelle (7)	B-1900	Cold Press, Sinter, + Heat Treat	1900	103	14	96.5	15.2	2.4	50.2
HMI (8)	IN-100	Hot Press, Extrude	1800	980	25	172.3	21.1	4.5	48.1
TRW (9)	B-1900	Slip Cast, Sinter, Heat Treat	1800	980	25	172.3	36.9	5.7	48.6
Nuclear Metals (10)	B-1900	Extrude, Heat Treat	1900	103	15	104	20.6	3.4	50.1
NASA (2)	713C	Extrude, Gradient Heat Treat	1800	980	15	104.0	1391.2	-	52.1
			1800	980	21	144.9	154.8	-	49.8
MIT (11)	IN-100	Extrude, Heat Treat	1800	980	12	82.7	170	-	50.0
			1800	980	15	104.0	75	-	49.4
			1800	980	20	138.0	12	-	47.6

(1) Larson Miller Parameter $P = T(20 + \log t)$.

Table 19

Tensile Properties of Superalloy Powder Metallurgy Alloys Compared to Cast Alloys

Source	Alloy	Condition	Temperature		Ultimate Strength		Yield Strength		Elongation %
			(°F)	(°C)	ksi	MN/m ²	ksi	MN/m ²	
NASA (3)	TRW-NASA-VIA	As Extruded	1800	980	13.5	93.0	-	-	220.0
			2000	1090	2.8	19.3	-	-	>300.0
		Extruded Heat Treat	2000	1090	1.5	10.4	-	-	160.0
NASA (2)	713C	Extruded Heat Treat	1800	980	70.5	486	-	-	20.0
Stellite Div. Cabot (4)	IN-100	Extruded Heat Treat	1800	980	63.9	441	37.1	257	9.2
Federal Mogul (5)	U-700	Extruded Heat Treat	1800	980	55.0	379	48.0	331	33.0
			1800	980	30.0	207	26.0	180	-
			1900	1038	20.0	138	-	-	5.0
Inco Data Brochure	IN-100	Cast	1800	980	82.0	565	54.0	372	6.0
			1900	1038	65.0	448	40.0	276	-
	U-700	Bar	1800	980	52.0	358	44.0	303	28.0
	713C	Cast	1800	980	68.0	469	44.0	303	20.0
			1800	980	68.0	469	44.0	303	20.0
NASA (3, 12)	TRW-NASA-VIA	Cast	1875	1075	71.0	490	61	421	5.5
			2000	1090	49.0	338	45	310	4.8

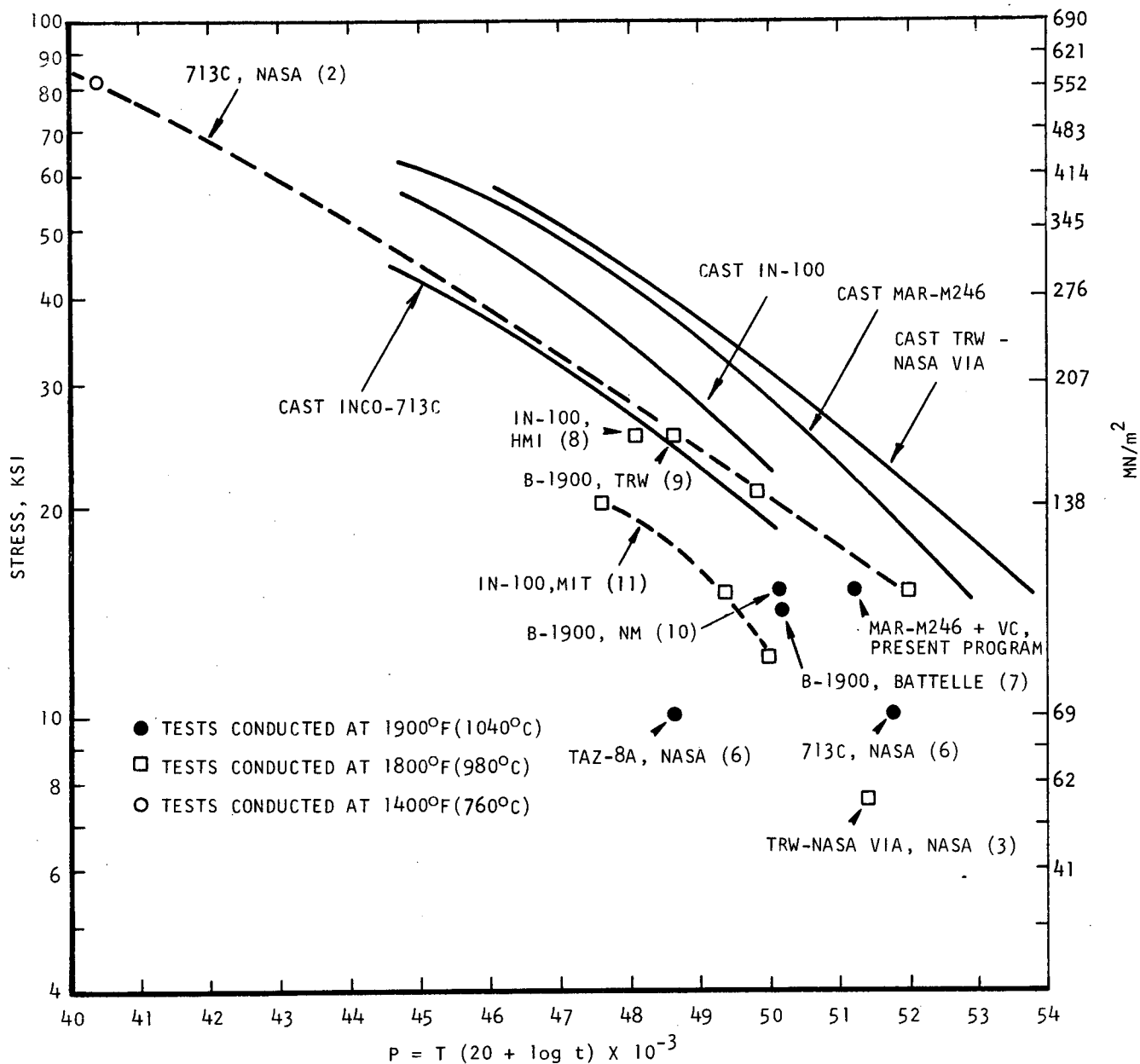
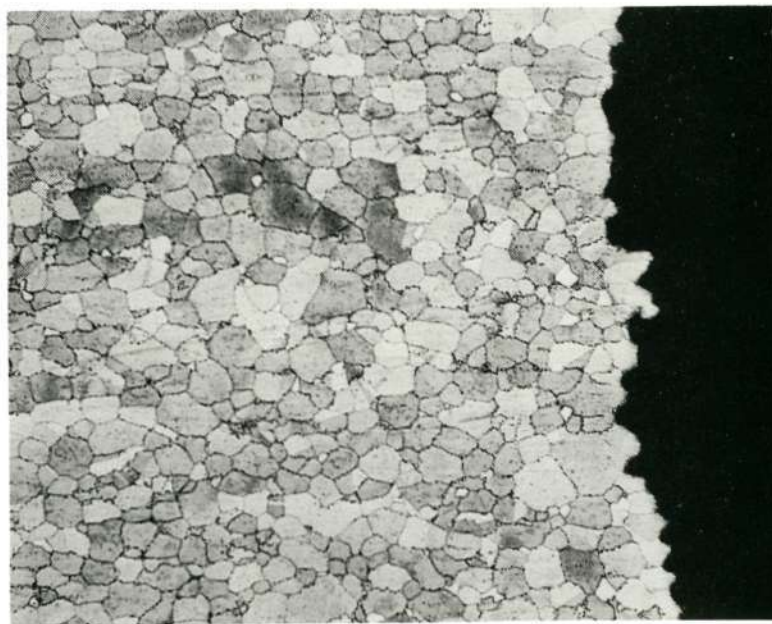


Figure 25. Stress rupture properties of base MAR-M246 powder alloy plus VC (0.28 w/o C) cast superalloys and powder metallurgy alloys of comparable composition.



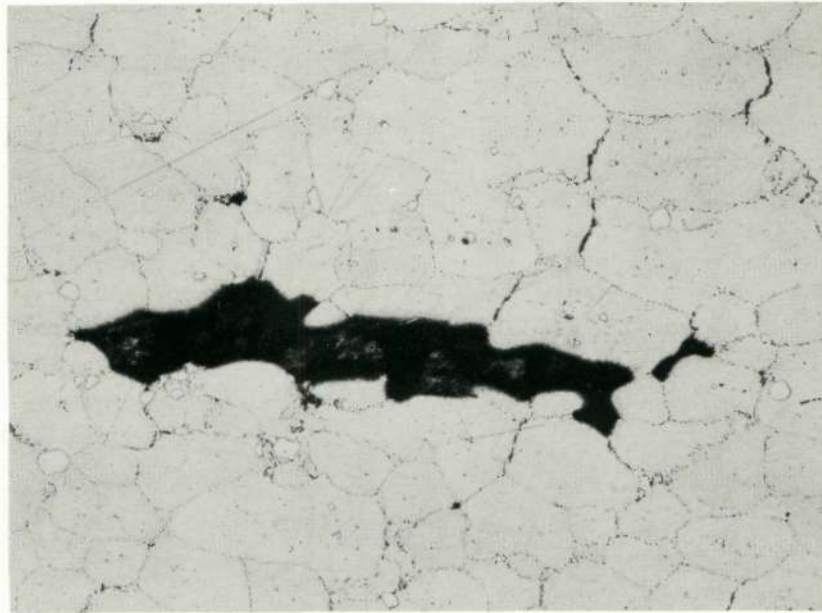
a) Base alloy + VC (0.15 w/o C), 12.4 hours life, ASTM grain size No. 7.



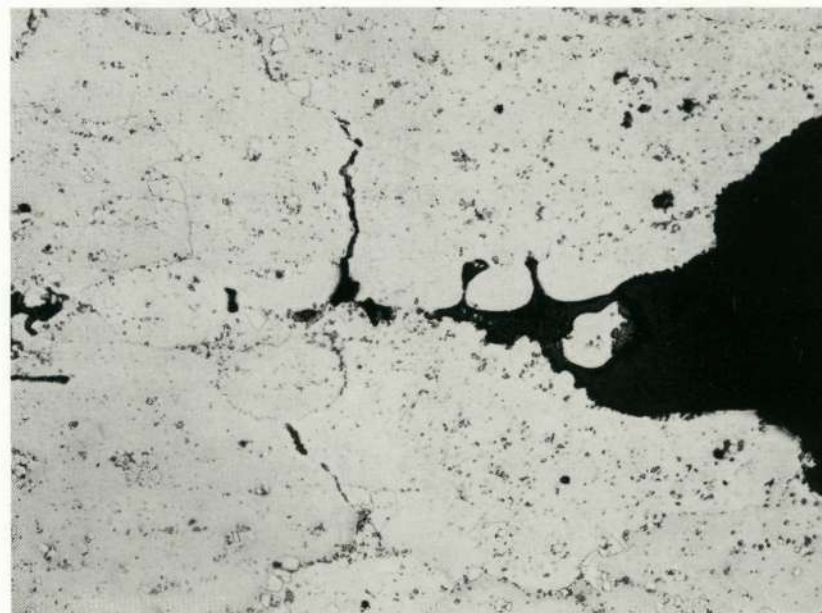
b) Base + VC (0.28 w/o C), 47.9 hours life, ASTM grain size No. 2-4.

This page is reproduced at the back of the report by a different reproduction method to provide better detail.

Figure 26. Light photomicrographs of 1900°F (1040°C) stress rupture test bars from second alloy series. Heat treatments and complete stress rupture results are listed in Table 16. All are longitudinal sections, 100X, etched.



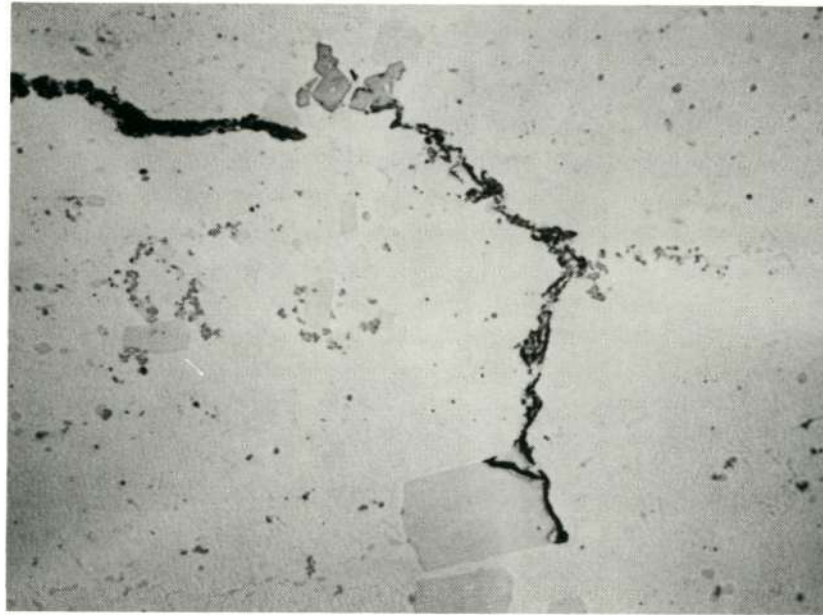
a) Base alloy + VC (0.15 w/o C), 22.1 hours life, ASTM grain size No. 3-4.



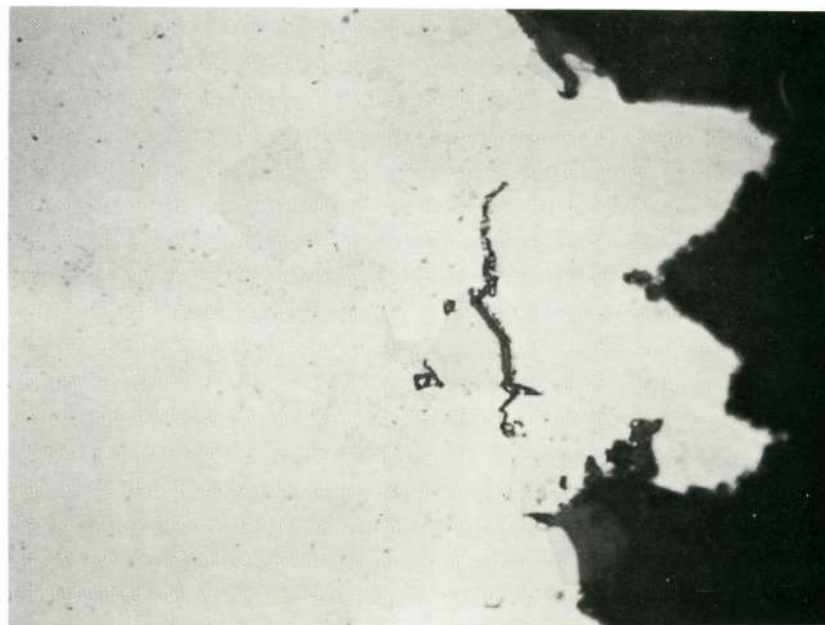
b) Base alloy + VC (0.28 w/o C), 26.3 hours life, ASTM grain size No. 1-2.

This page is reproduced at the back of the report by a different reproduction method to provide better detail.

Figure 27. Light photomicrographs of 1900°F (1040°C) stress rupture bars from second alloy series showing areas of incomplete compaction. Heat treatment and complete stress rupture results are listed in Table 16. All are longitudinal sections, 100X, etched.



a) Base alloy + VC (0.28 w/o C), 51.7 hours life, unetched.



b) Base alloy + TiC (0.20 w/o C), 25.5 hours life, etched.

This page is reproduced at the back of the report by a different reproduction method to provide better detail.

Figure 28. Light photomicrographs of 1900°F (1040°C) stress rupture bars from second series alloys showing crack emanating from angular carbide phase. Heat treatment and complete stress rupture results are listed in Table 16. All are longitudinal sections, 500X.

containing VC and TiC. Microprobe analysis of the angular carbides in the grain boundary region was conducted for the base alloy plus VC and the results are shown in Figure 29. The quantitative microprobe analysis for the angular carbide is listed in Table 20. These results indicated that the original VC decomposed during consolidation and heat treatment to a tantalum and titanium rich carbide plus the angular carbide phase consisting primarily of tungsten, nickel, molybdenum, and chromium.

Attempts to thermally decompose the angular carbides were unsuccessful except for the low carbon content base alloy plus VC (0.15 w/o C). An exposure for 100 hours at 1700°F (925°C) applied to the failed stress rupture bars produced a modification of the morphology from an angular shape to a rounded shape, Figure 30. This morphology was considered less detrimental to high temperature properties. X-ray diffraction analysis of extracted residues from these specimens was conducted to determine the nature of acicular phases observed to form throughout the grains after the 100 hour treatment at 1700°F (925°C), Figure 30a. For all three alloys, MC type carbides were detected as well as M₆C type carbides. The M₆C indications for the base alloy plus TiC (0.20 w/o C) were not as definite as for material containing VC, however. M₆C type acicular phases were also observed in the nickel-base superalloy B-1900 after long time thermal exposures (13) but were not detrimental to high temperature properties. It is believed that the 1700°F (925°C) aging treatment should be beneficial to high temperature properties by altering the morphology of the angular carbide phase and that the accompanying formation of acicular M₆C carbides should not adversely affect these properties.

In addition to these three microstructural features, metallographic examination also revealed that sufficient decomposition of the original carbides occurred during the subsequent aging treatment to allow the precipitation of discrete particle MC carbides along the grain boundaries. An example of this morphology is shown in Figure 31.

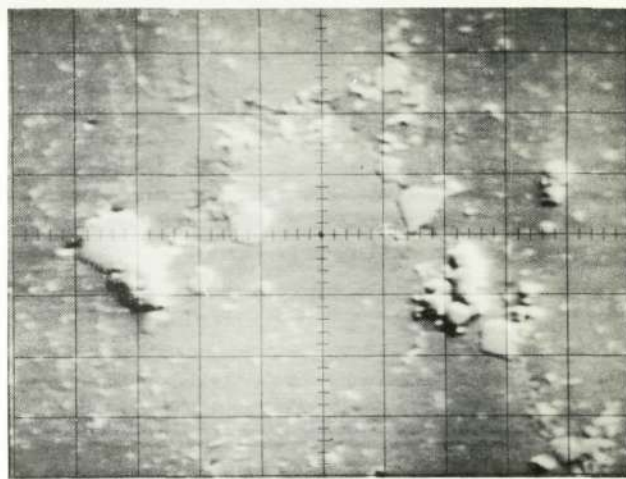
3. Summary - Second Alloys Series

The purpose of evaluating the three extrusions in the second alloy series was to optimize the carbon content in the material. In addition, the alloy containing TiC (0.20 w/o C) was used to compare the resultant mechanical properties of argon versus hydrogen atomized powder. The base alloy plus VC (0.28 w/o C) exhibited the best combinations of stress rupture and tensile properties. The hydrogen-atomized base alloy plus TiC (0.20 w/o C) exhibited slightly better stress rupture and tensile properties than its argon-atomized counterpart.

Metallographic analysis of the failed test bars revealed that three microstructural features may have contributed to premature failure. First, grain size variation from specimen to specimen was observed in spite of the fact that thermal mechanical working operations were conducted to



a) Light photomicrograph, 500X.



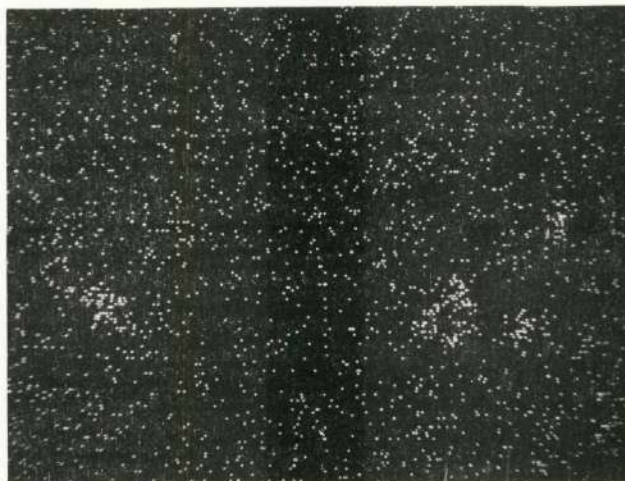
b) Back scattered current image, 325X.



c) W raster, 325X



d) Ta raster, 325X



e) Ti raster, 325X

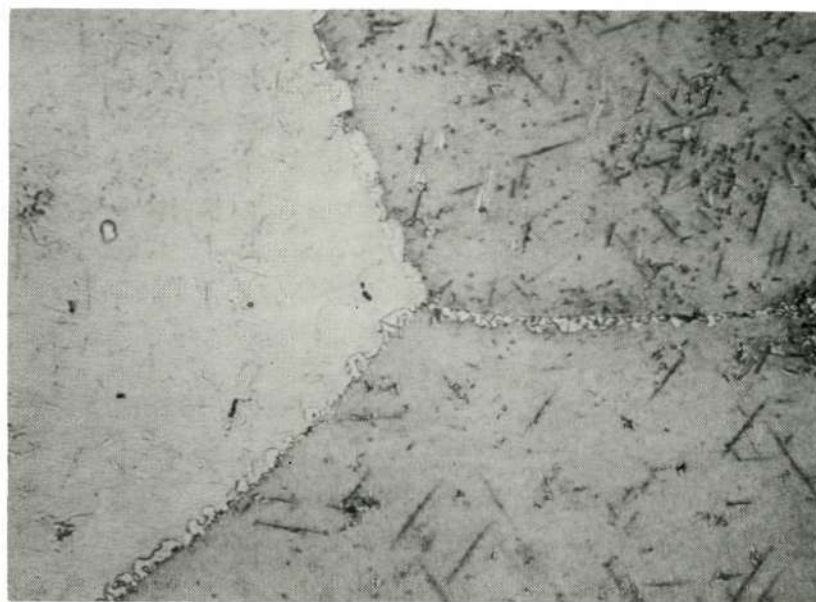
This page is reproduced at the back of the report by a different reproduction method to provide better detail.

Figure 29. Microprobe examination of angular carbide phase in second series base alloy plus VC (0.28 w/o C), 1900°F (1040°C)/15 ksi (104 MN/m²) stress rupture specimen, 47.9 hours life.

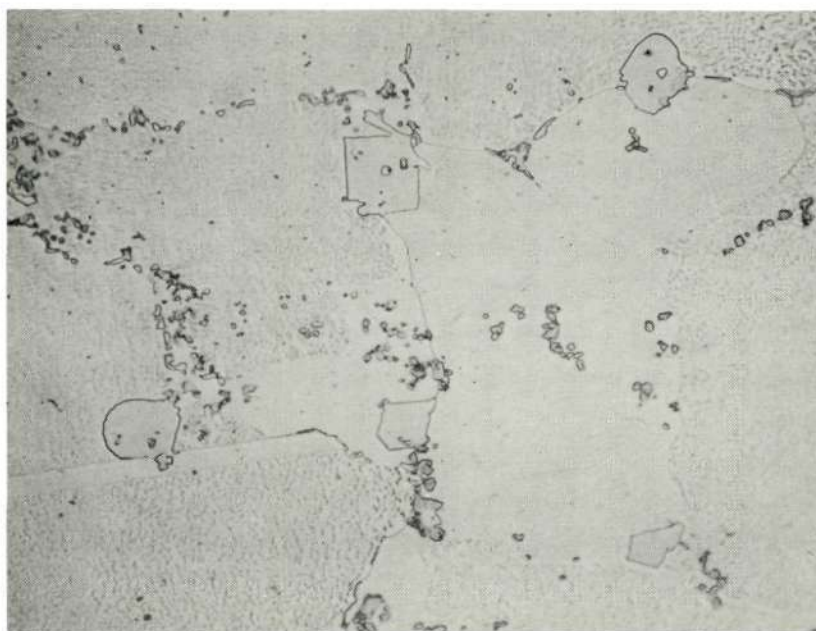
Table 20

Electron Microprobe Analysis of Angular Phase Observed in
Base Alloy + VC (0.28 w/o C)

<u>Element</u>	<u>Weight Percent</u>	<u>Atomic Percent</u>
W	56.53	23.87
Ni	13.31	17.61
Mo	8.23	6.65
Cr	6.43	9.61
Co	4.73	6.26
C	4.73	30.34
Ta	3.30	1.42
V	1.82	2.78
Ti	0.90	1.46



a) Base alloy + VC (0.15 w/o C) 2.9 hours rupture life, + 100 hours at 1700°F (925°C).



b) Base alloy + VC (0.28 w/o C) 26.3 hours rupture life + 100 hours at 1700°F (925°C).

Figure 30. Light photomicrographs of second alloy series failed stress rupture bars. Each had 100 hours at 1700°F (925°C) aging treatment to alter morphology of deleterious angular carbides. Complete stress rupture results listed in Table 16. All are longitudinal sections, 500X, etched.

This page is reproduced at the back of the report by a different reproduction method to provide better detail.



Base alloy + VC (0.28 w/o C), 47.9 hours
life.

This page is reproduced at the
back of the report by a different
reproduction method to provide
better detail.

Figure 31. Light photomicrograph of second alloy series base alloy + VC (0.28 w/o C) after 1900°F (1040°C) stress rupture tests showing discrete particle grain boundary precipitation. Complete stress rupture results are listed in Table 16. Longitudinal section, 500X, etched.

impart controlled amounts of deformation into the microstructure. In general, specimens exhibiting the best stress rupture lives also had the largest grains. In most instances, however, these grain sizes were not identical to those obtained from heat treatment specimens. Second, incomplete compaction resulted from utilizing the larger mesh size hydrogen-atomized powder. This porosity, while unlike that produced in the heat treated argon-atomized material, was still detrimental to high temperature properties. Third, an angular carbide resulting from the decomposition of the original blended carbides was observed to contribute to premature failure by initiating cracks. It was possible, however, to mitigate the crack initiating tendency of this microconstituent by low temperature aging heat treatments.

C. Series 3 - Elimination of Microstructural Defects

As a result of the mechanical property and metallographic analysis of the three second series alloys, it was decided to optimize properties in a final extrusion by elimination of incomplete compaction and modification of the embrittling angular MC carbide. The base alloy plus VC (0.28 w/o C) was chosen for this extrusion because it exhibited the best stress rupture and tensile properties and most desirable discrete particle type grain boundary precipitation.

1. Materials and Procedures

Powder consolidation to eliminate incomplete compaction was conducted in a two step operation consisting of hot pressing followed by hot extrusion. Prior to hot pressing, powders were weighed out according to the formulation presented in Table 12 for extrusion No. 8 and blended in a nitrogen atmosphere for two hours, canned under a protective covering of argon, vacuum washed at 1000°F (540°C) for 24 hours and then pressed in the extrusion press at 2175°F (1190°C) using a blank die. The hot pressed billet was then turned down, recanned and extruded at a 16:1 reduction ratio at 2175°F (1190°C), a ram speed of 2.1 inches (5.3 cm)/second and a breakthrough pressure of 115 ksi (793 MN/m²). As will be discussed later, even this two step process did not completely compact this material.

The extruded bar stock was inspected utilizing procedures described previously, sectioned into 3 inch (7.6 cm) lengths, side press forged in the 150 ton Lake Erie hydraulic press, fully heat treated including modification of aging treatments to alter the morphology of the deleterious angular carbide phase and evaluated for mechanical property tests including room temperature, 1400°F (760°C), and 1900°F (1038°C) tensile tests and 1900°F (1038°C) 15 ksi (104 MN/m²) stress rupture tests. Light metallographic analysis was conducted on all failed bars to determine locations of fracture initiation.

2. Results and Discussion

a. Radiographic and Chemical Analysis

X-ray analysis indicated that the extrusion was completely free of macrodefects. Results of the chemical analysis of the extruded material are listed in Table 21. The carbon content varied from 0.17 w/o to 0.24 w/o and was rechecked from material taken from several locations in the extrusion. This degree of variation in carbon content suggested that the blending technique employed in this program did not promote sufficient homogeneity throughout the microstructure as far as carbon was concerned and was also responsible for the inconsistent grain growth heat treatment response observed throughout the program.

b. Solution and Grain Growth Heat Treatment Study

In order to verify that an ASTM No. 2-3 grain size could be attained in this alloy, material was side pressed at 2150°F (1175°C) at a 5% reduction in diameter and given various solution heat treatments. All solution heat treatments were conducted in a tube furnace under a protective argon atmosphere. A heat treatment was selected consisting of 24 hours at 2380°F (1305°C) plus 24 hours at 2390°F (1310°C) prior to the 2400°F (1315°C) exposure to provide homogenization in the grain boundary areas and resulted in the desired ASTM No. 2-3 grain size.

On the basis of previous work, the solution heat treatment was followed by a 1975°F (1080°C) age for 4 hours and a 1700°F (925°C) age for 100 hours.

c. Mechanical Property Results

The mechanical property results for this last extrusion including 1900°F (1038°C)/15 ksi (104 MN/m²) stress rupture and room temperature, 1400°F (760°C), and 1900°F (1038°C) tensile properties are listed in Table 22. Instead of the three step solution heat treatment selected for this alloy, a two step treatment omitting the 24 hour exposure at 2400°F (1315°C) was used because the test bar preform surface condition after the first two steps indicated that incipient melting would occur after the higher exposure. A metallographic specimen cut from one of the unmachined test bars did exhibit triple point incipient melting after being exposed at 2400°F (1315°C).

Although the stress rupture lives were consistent, they were below those for the singly extruded similar alloy, Table 16. The room temperature and 1400°F (760°C) tensile strengths of double compacted material were comparable to that of the singly extruded alloy, but the 1900°F (1038°C) strength properties were slightly lower, Table 17. In all instances the ductility values of the double compacted material were superior to those of the single extruded material, particularly at 1900°F (1038°C), and reflect the fact that the 1700°F (925°C) age for 100 hours was successful in mitigating the crack initiation tendencies of the angular MC carbide.

Table 21

Chemical Analysis of Third Series Extruded Billet

<u>Element</u>	<u>Aim</u>	<u>Analysis</u>
C	0.28	0.17, 0.23, 0.24*
Si	-	0.12
Cr	9.0	8.98
Mo	1.5	1.47
Al	5.2	5.0
Hf	0.6	0.58
V	1.8	1.35
O ₂	0.095	0.019
W	10.0	10.6
Co	9.0	8.93
Ti	1.6	1.46
B	0.015	0.011
Zr	0.05	0.085
Ta	1.7	1.69
Ni	Balance	Balance

* Recheck on the C analysis

Mechanical Property Results for Third Series Alloy
Base + VC (0.28 w/o C)

Temperature (°F)	(°C)	Ultimate Strength (ksi)	MN/m ²	Yield Strength (ksi)	MN/m ²	Elongation %	R.A. %
Room	22	124.3	857	110.0	758	2.9	3.7
1400	760	135.3	933	120.6	832	2.1	4.3
		114.5	789	110.7	762	1.3	1.6
1900	1038	40.2	277	33.5	231	5.3	6.6
		42.1	291	34.8	240	5.7	5.9

Room	22	140	965	125	862	5.0	-
1400	760	150	1040	125	862	5.0	-
1800	980	80	552	55	379	8.0	-
1900(3)	1038	62.5	431	37.0	255	-	-

<u>Life</u>	<u>Elongation %</u>	<u>R.A. %</u>	<u>ASTM Grain Size</u>	<u>Larson-Miller Parameter (4)</u>
17.7	4.3	4.3	6	49.5
18.3	4.8	3.9	5	49.5
15.9	3.1	6.3	6	49.2

100	2-3	51.9
-----	-----	------

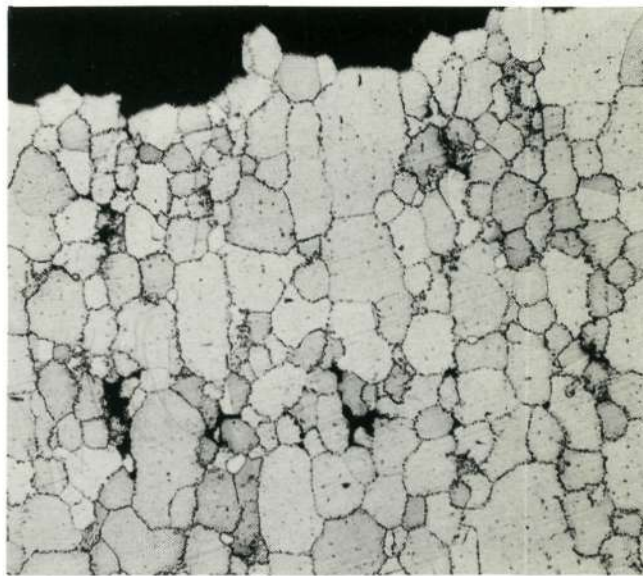
- 85

d. Metallographic Analysis of Failed Test Bars

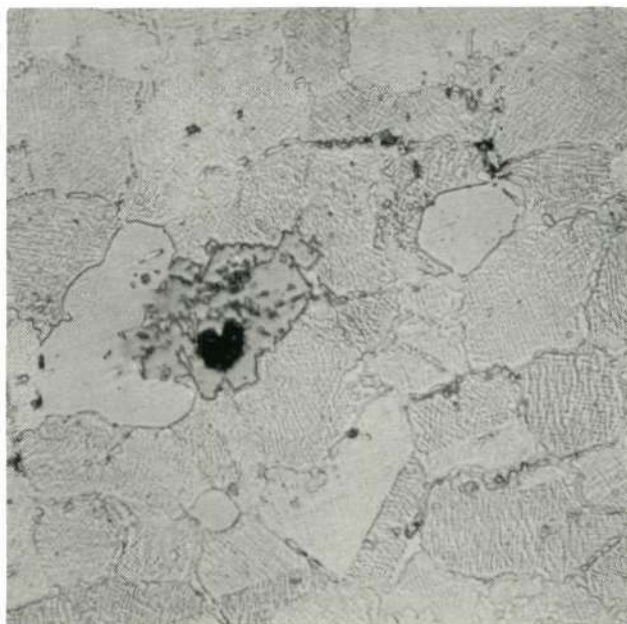
Metallographic evaluation revealed that the primary microstructural features responsible for the property levels exhibited by the double compacted material were the fine grain size and the persistent presence of incomplete compaction. As shown in Figure 32a, the specimen with 17.7 hours rupture life had a grain size of ASTM No. 6. Poor compaction is shown in Figure 32b for the specimen exhibiting a 1400°F (760°C) ultimate tensile strength of 135.5 ksi (943 MN/m²). That the desired discrete particle grain boundary carbide precipitation was maintained and that rounding of the angular carbide phase was achieved are indicated in Figure 32c for the specimen with 17.7 hours rupture life.



a) 17.7 hours rupture life, 100X ASTM grain size No. 6.



b) 1400°F (760°C) ultimate strength - 135.5 ksi, 100X, ASTM grain size No. 3-6, incomplete compaction.



c) Base alloy + VC (0.28 w/o C), 17.7 hours rupture life. Discrete grain boundary carbides and rounded MC carbides, 500X.

Figure 32. Light photomicrographs of final extrusion, base alloy plus VC (0.28 w/o C), failed test bars. All are longitudinal sections, etched. Heat treatments and property results are listed in Table 22.

This page is reproduced at the back of the report by a different reproduction method to provide better detail.

VI CONCLUSIONS

The objective of this program was to develop a nickel-base superalloy for 1800°F (980°C) - 2000°F (1090°C) temperature applications. The program was divided into two tasks. Task I consisted of powder production and consolidation while Task II consisted of property optimization. As a result of this program the following conclusions are offered:

1. The feasibility of a unique concept for alloying carbon into a superalloy powder matrix and achieving both grain growth and a discrete particle grain boundary carbide precipitation was demonstrated. The process consisted of blending metastable carbides with a carbon free base alloy and consolidating this blend by hot extrusion. This was followed by heat treating the consolidated material to grow a desired ASTM No. 2-3 grain size and to solution the metastable carbides to allow precipitation of discrete particle grain boundary carbides during subsequent aging heat treatments.
2. The best alloy developed during this program was hydrogen-atomized, thermal-mechanically processed modified MAR-M246 base alloy plus VC (0.28 w/o C). In the as-extruded condition the ultimate tensile strength at room temperature and 1400°F (760°C) exceeded that of the cast alloy by 57% and 34%, respectively, but at 1900°F (1038°C) was 33% lower. At room temperature and 1900°F (1038°C) the percent elongation was 120% above while at 1400°F (760°C) was comparable to that of the cast alloy. The 1900°F (1038°C)/15 ksi (104 MN/m²) stress rupture life was appreciably below the 100 hour rupture life for MAR-M246 while the rupture ductility was eight times greater. In the fully heat treated condition the alloy exhibited a maximum 1900°F (1038°C)/15 ksi (104 MN/m²) stress rupture life approximately 52% of that for cast MAR-M246, a 1900°F (1038°C) tensile strength approximately 78% of that for cast material, and room temperature and 1400°F (760°C) tensile strengths approximately 90% and 76%, respectively, of cast MAR-M246. In all instances the ductilities after heat treatment for high temperature properties were only 40% of their cast alloy counterparts. With the possible exception of 1800°F (980°C) results recently reported by NASA for Inco 713C (2), these properties are the best combination offered by conventional powder metallurgy processing to date.

3. Metallographic analysis of failed test bars revealed three microstructural defects the elimination of which should provide a considerable increase in the property levels obtained in this program. First, the blending operation utilized to mix the original metastable carbides with the base powder did not produce a homogeneous carbide distribution throughout the microstructure. Second, incomplete compaction as a result of utilizing the large mesh size hydrogen atomized powder resulted in porosity in the microstructure. Third, was the formation of an angular crack inducing carbide which resulted from the decomposition of the original blended metastable carbides. Although this morphology was altered by the use of a 100 hour 1700°F (925°C) aging treatment, a more optimum condition should occur if this carbide were smaller and more evenly distributed throughout the matrix.
4. Although the presence of argon was not definitely established, substitution of hydrogen-atomized powder for argon-atomized powder eliminated the occurrence of gas porosity observed in specimens heat treated at temperatures above 2250°F (1232°C). Although the presence of argon may not be detrimental to the properties of argon atomized powder material if heat treated below a critical temperature gas porosity was observed in specimens of argon atomized material heat treated above 2250°F (1232°C). This type of porosity was not observed in material produced from hydrogen-atomized powder and its elimination was a major factor contributing to the high property levels achieved in this program.

VII RECOMMENDATIONS FOR FUTURE WORK

In this program, a unique powder metallurgy process was proven feasible for the addition of carbon into a superalloy matrix which permitted both grain growth and discrete particle grain boundary carbide precipitation at the grain boundaries. The best combination of properties were, compared to cast MAR-M246, 52% of its 1900°F (1038°C)/15 ksi (104 MN/m²) stress rupture life, 78% of its 1900°F (1038°C) tensile strength, 76% of its 1400°F (760°C) tensile strength, 90% of its room temperature tensile strength and 40% of its ductility at these temperatures. Although these results represent one of the best combinations of high temperature properties reported thus far for material produced by conventional powder metallurgy processing, metallographic analysis revealed that the process techniques were not completely optimized.

The process of blending metastable carbides with the carbon free base powder allowed carbon to be added to the alloy without impairing grain growth. Decomposition of these carbides was effective in producing the desired discrete particle type grain boundary carbide precipitation during subsequent heat treatments. Several problem areas were defined, the solutions to which should considerably improve the property levels exhibited by these alloys.

For example, incomplete compaction was observed in the consolidation process consisting of hot pressing followed by hot extrusion. In order to avoid incomplete compaction, a process such as hot isostatic pressing should be investigated as an alternative consolidation technique. This process has been used to successfully consolidate hydrogen-atomized powder into fully dense bar stock with no evidence of internal porosity. In addition to this, the potential exists that the time, temperature, and pressure parameters for this process can be controlled so that a large grain size can be produced directly in the consolidated material possibly eliminating the need for complex thermal mechanical processing techniques to produce the required ASTM No. 2-3 grain size.

In addition to the incomplete compaction problem, mixing the powder constituents dry in a conical blender did not produce a homogeneous carbide dispersion throughout the alloy. This resulted in carbon variations throughout the extruded bar stock and an inconsistent grain growth heat treatment response. In order to develop a more uniform distribution of metastable carbides throughout the microstructure a process such as mechanical alloying should be investigated as an alternative blending technique. The process involves the recurrent cold welding of the alloy constituents to the ball surfaces of a dry high energy ball mill (Attritor) and flaking off of the composite aggregates until all of the constituents are finely divided and uniformly distributed through the interior of each powder particle (14,15).

Mechanical alloying should also mitigate the problems experienced with the large embrittling angular MC carbides resulting from the thermal decomposition of the added metastable carbides. A small size of the metastable carbides should enhance their thermal decomposition and minimize the formation of massive angular MC carbides throughout the microstructure. Treatments similar to the 100 hour exposure at 1700°F (925°C) can then be used to alter the undesirable angular morphology of these particles and blunt their crack initiating tendencies.

The success achieved by NASA with powder metallurgy Inco 713C (2) suggests that a temperature gradient annealing technique could also provide considerable property improvements in material blended with metastable carbides. By combining the long columnar grained microstructure resulting from the NASA process with the ability to precipitate discrete particle grain boundary carbides through the use of blended metastable carbides, strength levels should be enhanced considerably.

Finally, the program results suggest that chemical modifications also offer potential for improved high temperature properties. For example, carbon content was shown to have an important effect upon stress rupture life. Material containing VC at the 0.28 w/o carbon level offered the best combination of high temperature stress rupture and tensile properties, and a definite improvement was observed as carbon increased from 0.15 w/o to 0.28 w/o. In addition to carbon modifications, this powder metallurgy process also offers the potential to tailor the entire alloy chemistry to provide improved high temperature properties. Carbide formers, gamma-prime formers, and solid solution hardeners, for instance, can all be added without the segregation problems experienced with casting alloys. It is believed that a systematic evaluation of all these strengthening mechanisms, similar to that conducted in cast and wrought alloy development programs, would offer a significant contribution towards improved materials for gas turbine engine applications.

VIII REFERENCES

1. O. Kubaschewski, et. al., Metallurgical Thermochemistry 4th Ed., Pergamon Press, (1967).
2. R. V. Miner, "Study of a Columnar Grain Structure Produced in a Superalloy by Gradient-Annealing Prealloyed Powder Extrusions," NASA Technical Memorandum NASA TM X-2545, (April 1972).
3. J. C. Freche and R. L. Ashbrook, "Application of Powder Metallurgy to an Advanced Temperature Nickel-Base Alloy - NASA TRW VIA," NASA Technical Note TN D-6560, (November 1971).
4. Unpublished data from Stellite Division, Cabor Corp., (August 1971).
5. S. H. Reichman, B. W. Castledyne, J. W. Smythe, "Superalloy P/M Components for Elevated Temperature Applications," Automotive Engineering Conference, Detroit, Michigan, #700140, (January 12-16, 1970).
6. J. C. Freche, W. J. Waters and R. C. Ashbrook, "Evaluation of Two Nickel-Base Alloys, IN-713C and NASA TAZ-8A, Produced by Extrusion of Prealloy Powders," Metals Engineering Quarterly, (May 1970).
7. D. E. Kizer, "A Process for Making Sintered Articles from Superalloy Powders," Patent Application, Battelle Memorial Institute, (November 1970).
8. Private communication with J. Wentzel, Homogeneous Metals, Inc., (January 1972).
9. C. S. Kortovich, "Close Tolerance Forgings from Powder Metallurgy Preforms," AFML Final Report AFML-TR-69-181 prepared under Contract AF 33(615)-5411, (June 1969).
10. Unpublished TRW internal data, C. S. Kortovich.
11. L. N. Moskowitz, R. M. Pelloux, and N. J. Grant, "Properties of IN-100 Processed by Powder Metallurgy," Presented at the Second International Conference on Superalloys Processing, Champion, Pa., (September 18-22, 1972).
12. "TRW-NASA VIA - A High Strength Nickel-Base Cast Superalloy for Use at High Temperatures," TRW Data Brochure TM-4311, (August 15, 1967).
13. H. E. Collins, "The Effect of Simulated Jet Engine Thermal Conditions on the Microstructure and Mechanical Properties of Nickel-Base Superalloy B-1900, TRW Report No. TM-4591, (February 1971).

14. J. S. Benjamin and R. L. Cairns, "Elevated Temperature Mechanical Properties of a Dispersion Strengthened Superalloy," Paper presented at International P/M Conference, New York, N.Y., (July 12-16, 1970).
15. J. S. Benjamin, "Dispersion Strengthened Superalloys for Mechanical Alloying," International Nickel Report 649-OP, (September 16, 1969).